### **PCT**

# WORLD INTELLECTUAL PROPERTY ORGANIZATION International Bureau



## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification <sup>6</sup>:
A23G 3/30, A23L 1/22, 2/56, A61K 9/68

A1

(11) International Publication Number:

WO 99/13734

(43) International Publication Date:

25 March 1999 (25.03.99)

(21) International Application Number:

PCT/US97/16731

(22) International Filing Date:

18 September 1997 (18.09.97)

(71) Applicant (for all designated States except US): WM. WRIGLEY JR. COMPANY [US/US]; 410 North Michigan Avenue, Chicago, IL 60611 (US).

(72) Inventors; and

(75) Inventors; and
(75) Inventors; Applicants (for US only): WOLF, Fred, R. [US/US];
912 Muirhead Avenue, Naperville, IL 60450 (US). McGREW, Gordon, N. [US/US]; 2505 Ridge Road, Evanston,
IL 60201 (US). HOOK, Jeffrey, S. [US/US]; 3209 South
Clinton, Berwyn, IL 60402 (US). RICHEY, Lindell, C.
[US/US]; 1408 Eddy Lane, Lake Zurich, IL 60047 (US).
PATEL, Mansukh, M. [IN/US]; 3257 Venard Road, Downers Grove, IL 60515 (US). YATKA, Robert, J. [US/US];
15127 South St. Andrews Court, Orland Park, IL 60462
(US). WITKEWITZ, David, L. [US/US]; 7007 West 88th
Place, Bridgeview, IL 60455 (US). GREENBERG, Michal,
J. [US/US]; 1633 Brighton Court, Northbrook, IL 60062
(US). TYRPIN, Henry, T. [US/US]; 11732 Black Forest
Lane, Palos Park, IL 60464 (US). NELSON, Kathryn, T.
[US/US]; 838 West Altgeld #2, Chicago, IL 60614 (US).

(74) Agent: SHURTZ, Steven, P.; Brinks Hofer Gilson & Lione, Suite 3600, NBC Tower, 455 North Cityfront Plaza Drive, Chicago, IL 60611-5599 (US).

(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, HU, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).

#### Published

With international search report.

(54) Title: CHEWING GUM CONTAINING PHYSIOLOGICAL COOLING AGENTS

#### (57) Abstract

A method for producing a chewing gum, as well as the chewing gum so produced, incorporates a physiological cooling agent, such as acyclic carboxamide, or combinations of physiological cooling agents. In another embodiment a combination of physiological cooling agents is made in a modified release structure. The modified release/cooling agents combination is preferably obtained by physically modifying the properties of the combination of cooling agents by coating and drying. When incorporated into gum, these particles are adapted to enhance the shelf stability of the flavor and/or produce a modified release when the gum is chewed. In another embodiment, the physiological cooling agent is present with menthol and menthone. In another embodiment, coated chewing gum has a coating that comprises a physiological cooling agent. The preferred inventive chewing gum provides a high flavor impact in which the harsh notes normally associated with such a high flavor impact have been reduced or eliminated. In addition, the preferred inventive gum provides a clean, high-quality, cooling chewing gum coating with xylitol or other polyols where xylitol has been reduced in concentration or eliminated.

## FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	ES	Spain	I.S	Lesotho	Sī	Slovenia
AM	Armenia	FI	Finland	LT	Lithuania	SK	Slovakia
ΑT	Austria	FR	France	LU	Luxembourg	SN	Senegal
AU	Australia	GA	Gabon	LV	Latvia	SZ	Swaziland
ΑZ	A zerbaijan	GB	United Kingdom	MC	Monaco	TD	Chad
BA	Bosnia and Herzegovina	GE	Georgia	MD	Republic of Moldova	TG	
BB	Barbados	GH	Ghana	MG	Madagascar	TJ	Togo
BE	Belgium	GN	Guinea	MK	The former Yugoslav	TM	Tajikistan
3F	Burkina Faso	GR	Greece		Republic of Macedonia	TR	Turkmenistan
BG	Bulgaria	HU	Hungary	ML	Mali	TT	Turkey
ВЈ	Benin	12	Ireland	MN	Mongolia	UA	Trinidad and Tobago
BR	Brazil	IL	Israel	MR	Mauritania	UG	Ukraine
3Y	Belarus	IS	Iceland	MW	Malawi		Uganda
CA	Canada	IT	Italy	MX	Mexico	US	United States of America
F	Central African Republic	JP	Japan	NE	Niger	UZ	Uzbekistan
CG	Congo	KE	Kenya	NL	Netherlands	VN	Viet Nam
CH	Switzerland	KG	Kyrgyzstan	NO	Norway	YU	Yugoslavia
CI	Côte d'Ivoire	KP	Democratic People's	NZ	New Zealand	zw	Zimbabwe
CM	Cameroon		Republic of Korea	PL	Poland		
CN	China	KR	Republic of Korea	PT			
CU	Cuba	KZ	Kazakstan	RO	Portugal		
Z	Czech Republic	I.C	Saint Lucia	RU	Romania Russian Federation		
Œ	Germany	LI	Liechtenstein	SD	Russian receration Sudan		
K	Denmark	LK	Sri Lanka	SE.			
EE.	Estonia	LR	Liberia		Sweden		
		2310	Citeria	SG	Singapore		

### CHEWING GUM CONTAINING PHYSIOLOGICAL COOLING AGENTS

### Background of the Invention

5

10

15

20

25

The present invention relates to chewing gum compositions and methods of producing chewing gum. More particularly, the invention relates to producing chewing gum containing physiological cooling agents.

Preferably the physiological cooling agents are used in combination or have been treated to control their release and enhance shelf life stability. Also, the physiological cooling agents may be added individually or as part of a cooling flavor composition, or used in chewing gum coatings.

In recent years, efforts have been devoted to controlling release characteristics of various ingredients in chewing gum. Efforts have been directed at perfecting the use of high-intensity sweeteners within the chewing gum formulation, to thereby increase the shelf-life stability of the ingredients, i.e., the protection against degradation of the high-potency sweetener over time.

Patent Cooperation Treaty Publication No. 89-03170 discloses a method of controlling the release of acesulfame K. In this process, the sweetener is encapsulated fully or partially to modify the release rate in chewing gum.

Other patent publications disclose how a sweetener like aspartame can be physically modified to control its release rate in chewing gum.

For example, U.S. Patent No. 4,597,970 to Sharma et al. teaches a process for producing an agglomerated sweetener wherein the sweetener is dispersed in a hydrophobic matrix consisting essentially of lecithin, a glyceride and a fatty acid or wax having a melting point between 25 and 100°C. The disclosed method uses a spray congealing step to form the sweetener-containing matrix into droplets, followed by a fluid-bed second coating on the agglomerated particles.

10

15

- U.S. Patent Nos. 4,515,769 and 4,386,106, both to Merrit et al., teach a two step process for preparing a delayed release flavorant for chewing gum. In this process, the flavorant is prepared in an emulsion with a hydrophilic matrix. The emulsion is dried and ground and the particles are then coated with a water-impermeable substance.
- U.S. Patent No. 4,230,687 to Sair et al. teaches a process for encasing an active ingredient to achieve gradual release of the ingredient in a product such as chewing gum. The described method involves adding the ingredient to an encapsulating material in the form of a viscous paste. High shear mixing is used to achieve a homogeneous dispersion of the ingredient within the matrix, which is subsequently dried and ground.
- U.S. Patent No. 4,139,639 to Bahoshy et al. teaches a process of "fixing" aspartame by co-drying (by spray drying or fluid bed coating) a solution containing aspartame and an encapsulating agent, such as gum arabic, to thereby surround and protect the aspartame in the gum during storage.
- U.S. Patent No. 4,384,004 to Cea et al. teaches a method of encapsulating aspartame with various solutions of encapsulating agents using various encapsulation techniques, such as spray drying, in order to increase the shelf stability of the aspartame.
- U.S. Patent No. 4,634,593 to Stroz et al. teaches a method for producing controlled release sweeteners for confections, such as chewing gum. The method taught therein involves the use of an insoluble fat material which is mix mulled with the sweetener.

Several known compounds have what can be characterized as a

"cooling" activity, and are referred to in the art as "physiological cooling
agents." Physiological cooling agents are perceived as cold or cool when
contacted with the human body and, in particular, with the mucous
membranes of the mouth, nose and throat.

Efforts have been directed at perfecting the use of physiological cooling agents within chewing gum formulations to enhance flavor composition and control their release to enhance the flavor of chewing gum.

U.S. Patent No. 5,326,574 discloses a process for codrying the physiological cooling agent 3-l-menthoxypropane-1,2-diol with a food acceptable, water-soluble carrier and mixing the resulting product into chewing gum.

5

10

15

20

25

Peppermint oil is currently used to create a "cooling" in oral products such as toothpaste, mouthwash, chewing gum, candy and other food products. Peppermint oil generally comprises about 45% menthol, about 20% menthone, about 5% menthyl acetate, about 5% eucalyptol and many other constituents. Peppermint oil is even used in non-peppermint products, such as spearmint or wintergreen flavored products, in order to create this desired cooling effect. However, peppermint notes are then found in the resulting non-peppermint flavored products.

Menthol is also known for its physiological cooling effect on the skin and mucous membranes of the mouth. Being a major constituent of peppermint oil, menthol has been used extensively in foods, beverages, dentrifices, mouthwashes, toiletries, lotions and the like. The disadvantages of using menthol, however, are its strong minty odor and the harsh notes it imparts to compositions in which it is found.

A need, therefore, exists for a cooling flavor composition that will contribute a long-lasting cooling sensation to products in which it is found without the unwanted harshness or flavor characteristics that come from adding menthol.

It would be desirable to provide a high flavor impact chewing gum that does not manifest the harsh notes normally associated with some chewing gum. It would also be desirable to provide a clean, high-quality flavor chewing gum with a good cooling effect.

WO 99/13734

PCT/US97/16731

The present invention also relates to a coated chewing gum. Chewing gums are frequently covered with hard or soft coatings. The coatings provide an opportunity for the manufacturer to vary the taste, appearance, mouth-feel and nutritional value of the chewing gum.

5

10

For example, some consumers prefer a burst of intense flavor over the slow, gentle flavor release normally associated with chewing gum. In order to provide a favorable flavor impact, gum manufacturers have added flavors to the coating of a coated chewing gum. These flavors include spearmint flavor, peppermint flavor, wintergreen flavor and fruit flavors. In addition, very strong flavors such as menthol have often been used to provide a burst of flavor. However, at concentrations effective to provide a burst of flavor, menthol or mint flavors also manifest a bitter, harsh, burning taste (hereinafter "harsh notes").

The harsh notes are particularly acute with sugarless gum. In the case of coated gum with sugar, the sugar masks many of the harsh notes of menthol and mint flavors (although high levels of menthol or mint flavors can still manifest the harsh notes). However, recent efforts to produce sugarless coated gums have highlighted the difficulties of providing an initial burst of flavor without the harsh notes associated with traditional flavoring.

20

15

One method to alleviate the problem of harsh notes in sugarless coated chewing gum is the use of xylitol as a coating material. Xylitol has a sweetness level equivalent to sugar, and produces a cooling effect due to its endothermic heat of solvation. It produces a clean, high-quality flavor with a good cooling effect, particularly when it is used with menthol and mint flavors.

25

30

Coating with xylitol is described in U.S. Patent No. 4,105,801; U.S. Patent No. 4,127,677; U.S. Patent No. 4,681,766; U.S. Patent No. 4,786,511; and U.S. Patent No. 4,828,845.

The disadvantage of xylitol is that it is an expensive ingredient. Many efforts have been made to replace xylitol with a less expensive sugarless polyol. The most common and lowest cost polyol used in chewing gum is

10

25

30

sorbitol. However, panning with sorbitol has been very difficult since it is hygroscopic and does not readily crystallize.

A number of patents have been published that use various procedures to coat with sorbitol, including G.B. Patent No. 2,115,672; U.S. Patent No. 4,317,838; and U.S. Patent No. 4,753,790. A successful sorbitol hard coating was reported in U.S. Patent No. 4,423,086, particularly when the sorbitol was at least 99% D-sorbitol. However, the quality of coating never approached the quality of typical xylitol coatings.

Other coating patents that teach alternative polyols to xylitol include: U.S. Patent No. 4,840,797, which discloses a maltitol coating which required over 95% maltitol to obtain a good quality coating on pellet gum; U.S. Patents Nos. 5,248,508 and 4,792,453, which disclose a hydrogenated isomaltulose coating; and U.S. Patent No. 5,603,970, which discloses an erythritol coating.

Still other patents teach partially replacing the xylitol with sorbitol,

lactitol, or maltitol. However, these cannot be applied in the same solution,
but must be applied by alternating solutions. In other words, a solution of one
polyol is applied, then another solution of another polyol is applied. These
patents include: U.S. Patent No. 5,270,061; U.S. Patent No. 5,376,389; PCT
Application Nos. PCT/US93/09354 (published as WO95/08925);

20 PCT/US94/10406 (published as WO95/07625); and PCT/US93/087<del>3</del>0 (published as WO95/07622).

A booklet entitled "The Evaluation of Chewing Gum -- Xylitol and the Prevention of Dental Caries" published in 1985 by Xyrofin describes a coating formed by panning with a solution containing xylitol and up to 10% sorbitol. U.S. Patent No. 5,536,511 discloses a coating that comprises co-crystallized xylitol and erythritol. In an approach different from panning from a solution, U.S. Patent No. 4,146,653 discloses a molten blend of xylitol and sorbitol that are used to form a coating.

U.S. Patent No. 5,409,715 discloses coating chewing gum with various materials, including waxes, lipids, fatty acids, fats, oils, cellulose derivatives,

6

modified starch, dextrin, gelatin, zein, vegetable gums, proteins, edible polymers, edible plastic film, maltodextrins, polyols, low calorie carbohydrate bulking agents, shellac and combinations thereof.

It would be desirable to provide a high flavor impact chewing gum coating that does not manifest the harsh notes normally associated with such coatings. It would also be desirable to provide a clean, high-quality flavor coated chewing gum with a good cooling effect, while reducing or eliminating the xylitol currently preferred in sugarless coatings.

### Summary of the Invention

5

10

15

20

25

This invention incorporates a physiological cooling agent or combination of physiological cooling agents into a chewing gum. One preferred embodiment of the invention provides a chewing gum having a clean, cool sensation imparted by a cooling flavor composition that includes a physiological cooling agent. Another preferred embodiment also contains a flavor, and a combination of physiological cooling agents which have been treated so as to modify their release from the chewing gum. The result is a synergy between the physiological cooling agents and the flavor, which provides a high flavor impact at a lower concentration of flavor. Thus, with either or both aspects of the present invention, chewing gum can be made with a long lasting cooling sensation without unwanted harshness or flavor characteristics. The gum may have a high flavor impact, as well as a clean, high quality flavor with good cooling effect.

In the first aspect, applicants have discovered that using menthol and menthone in combination with one or more physiological cooling agents provides optimal cooling without unwanted flavor characteristics. Adding the physiological cooling agent provides the chewing gum with an unexpected, high-flavor impact where the harsh notes have been reduced or eliminated. This is particularly valuable for sugarless chewing gum where the harsh notes of the flavor are not masked by sugar.

In this first aspect, the invention is directed to cooling flavor compositions that provide the desired "cooling" sensation but not the unwanted harshness and flavor characteristics. The cooling flavor compositions of the present invention replace some or all of the peppermint oil presently used to create a "cooling." Because the cooling flavor compositions use menthol and menthone, as opposed to peppermint oil, the cooling flavor compositions do not add unwanted peppermint notes.

In addition, the physiological cooling agent provides a cooling sensation similar to that associated with gum made from xylitol.

Consequently, a lower cost polyol can be used as a chewing gum material without sacrificing the clean, high-quality, cooling sensation ordinarily associated with xylitol.

In one embodiment, the invention is a cooling flavor composition comprising:

15

10

5

- a) about 40% to about 80% by weight menthol;
- b) about 15% to about 50% by weight menthone; and
- c) about 3% to about 25% by weight of at least one physiological cooling agent selected from the group consisting of menthyl succinate; acyclic carboxamide; menthyl lactate; 3-l-menthoxypropane-1,2-diol; N-substituted p-menthane carboxamide; menthone glycerol ketals and mixtures thereof.

20

In another embodiment, the invention is an oral composition, such as chewing gum, comprising a flavor and a cooling flavor composition comprising:

- a) about 40% to about 80% by weight menthol;
- b) about 15% to about 50% by weight menthone; and
  - c) about 3% to about 25% by weight of at least one physiological cooling agent selected from the group consisting of menthyl succinate; acyclic carboxamide:

30

25

8

menthyl lactate; 3-l-menthoxypropane-1,2-diol; N-substituted p-menthane carboxamide; menthone glycerol ketals and mixtures thereof.

In this embodiment, the cooling flavor compositions are used in chewing gum to improve the "cool" sensation perceived upon chewing the gum and to extend the duration of the "cool" sensation.

5

10

15

20

25

30

In other embodiments of the invention, the cooling flavor compositions may also be used in other comestibles or even topical products such as creams and lotions.

In a second aspect, the present invention also includes a method for producing chewing gum with an acyclic carboxamide physiological cooling agent or combinations of physiological cooling agents, treated to have a modified-release. The controlled release combination of physiological cooling agents is obtained by modifying the cooling agents by encapsulation, partial encapsulation or partial coating, entrapment or absorption with water-soluble materials or water-insoluble materials. The procedures for modifying the physiological cooling agents include spray drying, spray chilling, fluid-bed coating, coacervation, extrusion, and other agglomerating and standard encapsulating techniques. The cooling agents may also be absorbed onto an inert or water-insoluble material. The cooling agents may be modified in a multiple step process comprising any of the processes noted.

The combination of cooling agents, or the combination of cooling agents when modified according to the present invention, give a chewing gum a controlled-release cooling agent. A higher quantity of cooling agents can be used without resulting in a high initial cooling agent impact, but instead having a delayed cooling release in chewing gum, giving a highly consumer-acceptable chewing gum product. Some cooling agents have a very slow release, but may be modified to give a fast release for more initial impact.

Another embodiment of this invention incorporates a physiological cooling agent into the coating of a coated chewing gum. One preferred

10

15

20

25

embodiment of the invention provides a chewing gum coating having a clean, coal sensation in which xylitol is replaced, in part or in whole, by a less expensive coating material.

Another preferred embodiment also contains a flavor, where the synergy between the physiological cooling agent and the flavor provides a high flavor impact at a lower concentration of flavor. Adding the physiological cooling agent provides the coated chewing gum with an unexpected, high-flavor impact where the harsh notes have been reduced or eliminated. This is particularly valuable for sugarless chewing gum where the harsh notes of the flavor are not masked by sugar.

In addition, the physiological cooling agent provides a cooling sensation similar to that associated with coatings made from xylitol. Consequently, a lower cost polyol can be used as a chewing gum coating material without sacrificing the clean, high-quality, cooling sensation ordinarily associated with xylitol coatings.

In yet another aspect of the invention, a chewing gum composition comprises an acyclic carboxamide physiological cooling agent and either menthol or another physiological cooling agent, or both.

In still another embodiment, a chewing gum composition comprises a hot flavor, such as cinnamon, and a physiological cooling agent. This embodiment produces a breath freshness perception.

The foregoing and other features and advantages of the present invention will become apparent from the following detailed description of the presently preferred embodiments, when read in conjunction with the accompanying examples.

#### **Brief Description of the Drawings**

FIG. 1 is a graph showing a rating of perception of fresh breath over time after chewing comparative and inventive gum samples.

WO 99/13734

10

PCT/US97/16731

FIG. 2 is a graph showing a rating of perceived breath odor over time after chewing comparative and inventive gum samples.

FIG. 3 is a graph showing a rating of perceived effectiveness for breath freshening over time after chewing comparative and inventive gum samples.

# Detailed Description of the Presently Preferred Embodiments of the Invention

5

10

15

20

25

In the context of this invention, chewing gum refers to chewing gum, bubble gum and the like. Moreover, all percentages are based on weight percentages unless otherwise specified. Further, although some terms are referred to in the singular, it is understood that such references may also encompass the plural. For example, although chewing gum coating is referred to in the singular, it is understood that coated chewing gum normally contains multiple layers of coating. Therefore a phrase that refers to "the coating," refers to one or more layers of coating. Finally, all references cited herein are incorporated by reference.

The composition of a chewing gum tends to suppress the release of its flavors. Although a slow flavor release is desirable in many instances, some consumers prefer a burst of intense flavor. One method to provide a chewing gum with a greater flavor impact is the addition of encapsulated flavor to a chewing gum. For example, for a cool and refreshing taste, cooling flavors such as encapsulated menthol and/or mint flavors are added to chewing gum. A menthol/mint combination is disclosed in U.S. Patent No. 4,724,151.

However, the improved flavor impact of adding cooling flavors to the chewing gum is somewhat offset by the disadvantage of the bitter, harsh, burning sensations associated with high concentrations of such flavors. This disadvantage is particularly acute for sugarless gum, since sugar tends to mask the harsh notes.

PCT/US97/16731

5

10

15

20

25

30

The inventors have found that adding a combination of physiological cooling agents or a combination of cooling agents that have a modified release from the chewing gum provides a favorable flavor impact. As a result, the inventors are able to reduce or eliminate the harsh notes associated with the prior art high flavor-impact chewing gums, even in the case of sugarless chewing gums.

By adding a combination of physiological cooling agents to a menthol or mint type flavored chewing gum, one can obtain a strong cooling and clean minty flavor, without the higher concentrations of menthol or mint flavors required in the prior art. Also, the fast release encapsulation of a combination of physiological cooling agents complement the mint flavors to give a high impact of flavor and cooling normally found in chewing gum. This cooling effect is like menthol cooling, but without the bitterness associated with menthol.

For sugarless gums, chewing gums containing xylitol have become very popular because xylitol has about the same sweetness level as sugar and a cooling ability due to its endothermic heat of solvation. With this sweetness, xylitol masks the harsh notes of high impact flavors such as menthol and mint flavors. At the same time, its cooling effect complements the cooling effect of the cooling flavors. As a result, xylitol provides a clean, high-quality cooling effect. Xylitol in combination with physiological cooling agents give chewing gum a high quality flavor profile with good cooling. However, because of its expense, various efforts have been made to replace xylitol in coatings with a less expensive ingredient, such as another polyol.

Another method to provide a chewing gum with a greater flavor impact is the addition of flavor to the coating of a coated chewing gum. For example, for a cool and refreshing taste, cooling flavors such as menthol and/or mint flavors are added to the coating of chewing gum.

However, the improved flavor impact of adding cooling flavors to the chewing gum coating is somewhat offset by the disadvantage of the bitter.

WO 99/13734

5

10

15

20

25

12

PCT/US97/16731

harsh, burning sensations associated with high concentrations of such flavors. This disadvantage is particularly acute for sugarless gum, since sugar tends to mask the harsh notes.

The inventors have found that adding a physiological cooling agent to the coating provides a favorable flavor impact. As a result, the inventors are able to reduce or eliminate the harsh notes associated with the prior art high flavor-impact coated chewing gums, even in the case of sugarless, coated chewing gums.

By adding a physiological cooling agent to a menthol or mint type flavored chewing gum coating, one can obtain a strong cooling and clean minty flavor, without the higher concentrations of menthol or mint flavors required in prior art coatings. Also, the physiological cooling agents complement the mint flavors to give a high impact of flavor and cooling normally found in coated chewing gum. This cooling effect is like menthol cooling, but without the bitterness associated with menthol.

Coated chewing gums of the present invention may be made with a variety of chewing gum compositions. The chewing gum is prepared as conventional chewing gum, but formed into pellets or balls. The pellets/balls can then be coated by a variety of methods known in the art, such as conventional panning methods to coat chewing gum. The coating isgenerally applied in multiple layers, where the composition of one layer is not necessarily the composition of the other layers.

The coating of the present invention contains at least a coating material and a physiological cooling agent. It may also contain other ingredients such as flavors, artificial sweeteners and dispersing agents, coloring agents, film formers and binding agents.

Coating material constitutes the substantial portion of the chewing gum coating. Examples of coating material include sugars such as sucrose, maltose, dextrose and glucose syrup; polyols such as maltitol, lactitol, xylitol,

mannitol, erythritol, sorbitol, hydrogenated isomaltulose and hydrogenated starch hydrolysates; and combinations thereof.

5

10

15

20

25

For sugarless gums, xylitol coatings have become very popular because xylitol has about the same sweetness level as sugar and a cooling ability due to its endothermic heat of solvation. With this sweetness, xylitol masks the harsh notes of high impact flavors such as menthol and mint flavors. At the same time, its cooling effect complements the cooling effect of the cooling flavors. As a result, xylitol provides a clean, high-quality cooling effect. When used with physiological cooling agents, a synergistic cooling effect may be noted with xylitol. However, because of its expense, various efforts have been made to replace xylitol in coatings with a less expensive ingredient, such as another polyol.

One problem with polyol substitutes for xylitol is that they are generally much less sweet than xylitol or sugar. In the presence of high levels of flavor, the polyol substitutes generally provide a coated product with a bitter, unpleasant taste. To some degree, high intensity sweeteners may be used to counteract bitterness, but these also may have some objectionable taste. Also, some of these polyols themselves may contribute to a bitter taste.

By using physiological cooling agents in a coating that comprises polyols other than xylitol, the coating manifests a cooling similar to xylitol cooling. Thus xylitol substitutes, which cost significantly less than xylitol, can be used to coat gum and give a taste sensation similar to xylitol.

Many of the coating material references cited above contemplate the addition of one or more flavors to chewing gum coating. However, none disclose the addition of a physiological cooling agent to give the coating a cooling sensation like that of xylitol.

By using a combination of physiological cooling agents treated to have a modified release in a chewing gum that comprises polyols other than xylitol, the effect is a cooling similar to xylitol cooling. Thus xylitol substitutes, which

PCT/US97/16731

cost significantly less than xylitol, can be used in gum and give a taste sensation similar to xylitol.

Physiological cooling agents encompasses any number of physiological cooling agents. However, in the context of this invention, the term "physiological cooling agent" does not include traditional flavorderivatives such as menthol or menthone. Preferred physiological cooling agents do not have a perceptible flavor of their own, but simply provide a cooling effect.

5

10

15

20

25

30

Since the physiological cooling agents do not have their own perceptible flavor, they can be used with other types of flavors to offer new and unique advantages, such as breath freshening. Most confectionery products which are promoted for breath freshening are mint flavored products which contain moderate to high levels of menthol. Menthol is a component of peppermint oil which has a cooling property which provides to the consumer a perception of freshness in the oral cavity.

Paradoxically, some cinnamon flavored products have also been marketed for breath freshening based on the physiological heating provided by cinnamic aldehyde and other spice flavor components. In some cases, menthol or peppermint oil has been added to cinnamon flavored products to provide a cooling sensation and enhance breath freshening. While-some consumers enjoy the presence of a mint note in cinnamon confections, others describe the flavor as "muddy", "dirty" and "confused", preferring a "pure" cinnamon flavor.

Another embodiment of the present invention is a confectionery product, preferably a chewing gum, which incorporates a spicy hot flavor, is substantially free of menthol and other mint oil components and which comprises a physiological cooling agent which imparts improved breath freshening and a desirable hot/cold sensory perception without a mint flavor.

It has surprisingly been found that many consumers, who may not appreciate the addition of mint flavor to cinnamon flavored candies and

chewing gums, do enjoy the addition of a non-menthol, physiological cooling agent in such products. Furthermore, it has been found that the inventive products have greatly enhanced breath freshening properties compared to cinnamon flavored confections without physiological cooling agents.

The confectionery products of this embodiment of the invention can be any new or existing type of product in that category. Hard candies, hard or soft pan coated items, chewy confections and powdered candies are specifically contemplated. Chewing gum is a preferred embodiment.

5

10

15

20

25

30

The confections of this embodiment of the invention will include a natural or non-menthol hot spice flavor such as cinnamon, clove, ginger, black pepper, cayenne pepper and mixtures of these. Cinnamon flavor is preferred. The flavor composition will be substantially free of menthol and mint oils. By substantially free, it is meant that the flavor composition will comprise less than about 15% and preferably less than 10% of mint components. Most preferably, the flavor composition will have 0% to 3% of mint components. The flavor composition will also contain a physiological cooling agent in an amount sufficient to impart noticeable cooling to the product.

While there are references that disclose the use of physiological cooling agents in chewing gum and other confections, unique combinations and controlled release are new areas of interest. Also, no references disclose the use of physiological cooling agents in a chewing gum coating. Because a flavor imparts a distinct and unique sensation when it is used in a chewing gum, some combinations of the physiological cooling agents offer unique advantages and may be combined with various types of flavors or with various methods of encapsulation and entrapment for controlled release.

Several U.S. and foreign references disclose specific compounds and classes of compounds that are physiological cooling agents that may be used in the present invention. Some of these disclose the use of physiological cooling agents in chewing gum. These include:

- U.S. Patent No. 5,451,404 (a ketal combined with another coolant (menthol or carboxamides));
- U.S. Patent No. 5,372,824 (physiological cooling agents and reduced menthol);
- 5 U.S. Patent No. 5,348,750 (menthone ketals);
  - U.S. Patent No. 5,326,574 (a spray dried 3-l-menthoxypropane-1,2-diol);
    - U.S. Patent No. 5,266,592 (menthone glycerol ketals);
- U.S. Patent No. 5,165,943 (a cyclodextrin complex with physiological cooling agents);
  - U.S. Patent No. 5,009,893 (p-menthane carboxamide physiological cooling agent with menthol for reduced bitterness);
    - U.S. Patent No. 4,459,425 (3-I-menthoxypropane-1,2-dioI);
    - U.S. Patent No. 4,296,093 (substituted cyclohexanamides);
- U.S. Patents Nos. 4,248,859 and 4,318,900 (alkyl substituted alicyclic carboxylic acids, esters or amides);
  - U.S. Patents Nos. 4,157,384 and 4,029,759 (various 3-substituted p-menthanes);
    - U.S. Patent No. 4,081,480 (alpha-oxy(oxo)mercaptan alkanes);
- 20 U.S. Patent No. 4,070,449 (sulphoxides and sulphones); -
  - U.S. Patents Nos. 4,060,091; 4,190,643 and 4,136,163 (substituted p-menthane-3-carboxamides):
  - U.S. Patents Nos. 4,153,679; 4,296,255 and 4,230,688 (acyclic carboxamides);
- U.S. Patent No. 4,034,109 (acyclic sulphonamides and sulphinamides);
  - U.S. Patent No. 4,033,994 (p-menthane-3-carboxylates);
  - U.S. Patents Nos. 3,793,446 and 3,644,613 (ketoesters of menthol):
  - U.S. Patent No. 3,720,762 (spilanthol with menthol or peppermint oil);

15

25

Canadian Patent No. 2,101,790 (carbonic acids having free polar groups);

German Patent No. 2,608,226 (menthyl lactate);

German Patent No. 2,433,165 (N-acetylglycine menthyl ester);

French Patent No. 2,577,922 (L-menthyl-3-hydroxybutyrate);

Japanese Patent No. 94 /065023 (2-isopropenyl-5-methylcyclohexanol);

Great Britain Patent No. 1,502,680 (bicyclic acids, esters, amides and substituted menthanols);

10 Great Britain Patent No. 1,476,351 (cyclic and acyclic amides, ureas and sulphonamides);

Great Britain Patent No. 1,442,998 (trialkyl-substituted cyclohexane carboxamides);

Great Britain Patents Nos. 1,421,744 and 1,421,743 (novel amides);

Great Britain Patent No. 1,411,786 (cyclohexanamides);

Great Britain Patent No. 1,404,596 (acyclic secondary and tertiary alkanols);

PCT Publication No. WO 97/07771 (menthyl succinate and carboxamides);

20 PCT Publication No. WO 96/28133 (coolant composition for – comestibles);

PCT Publication No. WO 96/17524 (a cooling composition comprising N-substituted p-menthane carboxamides and menthol);

PCT Publication No. WO 94/010117 (cyclohexanol derivatives); and U.S. Patent No. 3,639,569 (physiological cooling agents).

Particular examples of physiological cooling agents include:

 substituted p-menthanes, substituted p-menthane-carboxamides (e.g., N-ethyl-p-menthane-3-carboxamide (FEMA 3455)), acyclic carboxamides, substituted cyclohexanamides, substituted

- cyclohexane carboxamides, substituted ureas and sulphonamides, and substituted menthanols (all from Wilkinson Sword);
- 2) hydroxymethyl and hydroxyethyl derivatives of p-menthane (from Lever Bros.);
- 5 3) menthyl succinate:
  - 2-mercapto-cyclo-decanone (from International Flavors and Fragrances);
  - 5) 2-isopropanyl-5-methylcyclohexanol (from Hisamitsu Pharmaceuticals, hereinafter "isopregol");
- 10 6) hydroxycarboxylic acids with 2-6 carbon atoms;
  - 7) menthone glycerol ketals (FEMA 3807, tradename FRESCOLAT™ type MGA);
  - 8) 3-I-menthoxypropane-1,2-diol (from Takasago, FEMA 3784, (hereinafter "TCA"));
- 9) menthyl lactate; (from Haarman & Reimer, FEMA 3748, tradename FRESCOLAT™ type ML).

While any of the above-disclosed physiological cooling agents may be used in chewing gum, the presently preferred physiological cooling agents are:

- substituted p-menthane carboxamides (PMC), such as those disclosed in U.S. Patents Nos. 4,060,091; 4,190,643 and 4,136,163, all assigned to Wilkinson Sword, especially N-ethyl-p-menthane-3-carboxamide (called WS-3);
- 2) acyclic carboxamides (AC), such as those disclosed in U.S. Patents
   Nos. 4,296,255; 4,230,688; and 4,153,679; all assigned to
   Wilkinson Sword, especially N-2,3-trimethyl-2-isopropyl
   butanamide (called WS-23);
  - 3) menthone glycerol ketal (MGK);
  - 4) menthyl lactate (ML);
- 30 5) menthyl succinate (MS); and

10

15

20

25

30

#### 6) 3-l-menthoxypropane-1,2-diol (TCA).

The concentration of physiological cooling agent will depend on the intensity of the physiological cooling agent and the desired cooling effect. In general the concentration of cooling agents used is between about 0.001% and about 2% by weight of the chewing gum. The preferred concentration of cooling agent is between about 0.01% and about 1.0%, more preferably between 0.02% and about 0.5%.

The present invention contemplates that two or more physiological cooling agents may be added to the flavor used to make the chewing gum. On the other hand, the flavor and cooling agents may be added separately anywhere within the manufacturing process for making a chewing gum product.

These flavors include any flavor which is of food acceptable quality commonly known in the art such as essential oils, synthetic flavors or mixtures thereof. Such flavors include, but are not limited to, oils derived from plants and fruits such as citrus oils, fruit essences, peppermint oil, spearmint oil, eucalyptus, other mint oils, clove oil, oil of wintergreen, cinnamic aldehyde, anise and the like. Flavors that are very strong, such as menthol flavors, are also contemplated in this invention. Preferred flavors include cooling flavors such as peppermint, eucalyptus, menthol, wintergreen and fruity-mint; non-cooling flavors such as spearmint and cinnamon; and combinations thereof.

Artificial flavor components are also contemplated by the present invention. Those of ordinary skill in the art will recognize that natural and artificial flavors may be combined in any sensorially acceptable blend. All such flavors and blends are contemplated by the present invention.

The flavor may be added to the chewing gum formula in an amount such that it will contain from about 0.1% to about 10% flavor, preferably from about 0.2% to about 3.0% flavor, and most preferably about 0.5% to about 2% flavor.

5

10

15

20

25

30

For menthol flavored chewing gum, a combination of physiological cooling agents allows for a reduced overall concentration of menthol. However, it does not completely eliminate menthol, because menthol has a very identifiable unique taste and cooling sensation. Hence, in the case of menthol flavor, the physiological cooling agents only enhance the cooling while reducing the menthol bitterness.

The present invention thus is also directed to novel cooling flavor compositions comprising menthol and menthone that contribute a long-lasting cool sensation. These cooling flavor compositions may be substituted for peppermint oil in non-peppermint products, resulting in added coolness without harshness or unwanted flavor characteristics. They can, of course, also be used in peppermint flavored products.

It has been discovered that menthol, when used in conjunction with menthone, provides a cool sensation without the characteristically harsh tones of menthol. It has been further discovered that the use of menthol and menthone in combination with a physiological cooling agent results in a cooling flavor composition that provides initial optimal cooling, as well as extended coolness. The presence of only one of these components will not provide the desired results.

Menthol, which occurs naturally in peppermint oil, is the agent that provides the "cooling" sensation, but menthol alone tends to distort flavor notes. When used in conjunction with menthone, however, the result is cooling without harshness because the menthone helps to curb the harsh qualities of the menthol.

In one embodiment of the present invention, menthol and menthone are used in conjunction with one or more physiological cooling agents to produce a cooling flavor composition yielding optimal coolness. The menthol and menthone are used in their separated, or pure, form, as distinguished from being present in peppermint oil. Used as such, the menthol and menthone supply a fast release of the "cooling" sensation but do not add a

21

peppermint flavor. These cooling flavor compositions, which have a physiological cooling effect, can be used in food, drinks, dentrifices, gargles, cosmetics, lotions, etc. Suitable physiological cooling agents that are preferred in this aspect of the invention are menthyl succinate; menthyl lactate; 3-l-menthoxypropane-1,2-diol; menthone glycerol ketals; N-substituted p-menthane carboxamide; acyclic carboxamide and mixtures thereof. The preferred cooling agents are menthyl succinate, N-substituted p-menthane carboxamide (WS-3), acyclic carboxamide (WS-23) and menthyl lactate.

5

10

15

20

25

30

The cooling flavor composition comprising menthol, menthone and one or more physiological cooling agents is more potent than peppermint oil alone. As a result, less of the cooling blend is needed to achieve the desired coolness than peppermint oil. This would decrease the need for peppermint oil, a costly commodity which has an uncertain supply, saving money and conserving peppermint oil. However, if desired, peppermint oil may be added in addition to the cooling flavor compositions of the present invention.

In another embodiment, the cooling flavor composition comprising menthol, menthone and one or more physiological cooling agents is used in oral compositions such as chewing gum. The result is oral compositions having optimal cooling without harsh tones or unwanted flavor characteristics. Moreover, the blend of menthol, menthone and one or more synthetic cooling agents extends the coolness of the oral compositions in which the blend is used. This novel blend supplies a fast release of the cooling sensation from the menthol and menthone, and an extended release from the physiological cooling agents.

Because the cooling flavor compositions of the present invention will not impart an unwanted perpermint flavor to non-perpermint products, these compositions may be used to add coolness to fruit and other flavors.

Physiological cooling agents generally release slowly from chewing gum during the early stages of mastication of the gum because of their low

10

15

20

25

30

solubility in water. However, some like TCA are moderately fast release, others like WS-3, WS-23 are moderately slow release, and others like menthone glycerol ketal, menthyl lactate and menthyl succinate are very slow release.

Combinations of cooling agents may not only be synergistic but may also provide both moderate release and slow release to give flavor impact and flavor extension. Physical modifications of the physiological cooling agents by encapsulation with another substrate will also modify their release in chewing gum by modifying the solubility or dissolution rate. Any standard technique which gives partial or full encapsulation of the combination of physiological cooling agents can be used. These techniques include, but are not limited to, spray drying, spray chilling, fluid-bed coating, and coacervation. These encapsulation techniques that give partial encapsulation or full encapsulation can be used individually or in any combination in a single step process or multiple step process. Generally, a modified release of physiological cooling agents is obtained in multistep processes like spray drying the combined physiological cooling agents and then fluid-bed coating the resultant powder.

The encapsulation techniques here described are standard coating techniques and generally give varying degrees of coating from partial to full coating, depending on the coating composition used in the process. Also, the coating compositions may be susceptible to water permeation to various degrees. Generally, compositions that have high organic solubility, good film forming properties and low water solubility give better delayed release of the physiological cooling agents. Such compositions include acrylic polymers and copolymers, carboxyvinyl polymer, polyamides, polystyrene, polyvinyl acetate, polyvinyl acetate phthalate, polyvinyl pyrrolidone and waxes.

Although all of these materials are possible for encapsulation of physiological cooling agents, only food grade materials should be considered. Two standard food grade coating materials that are good film formers but not

water soluble are shellac and Zein. Others which are more water soluble, but good film formers, are materials like agar, alginates, a wide range of cellulose derivative like ethyl cellulose and hydroxypropylmethyl cellulose, dextrin, gelatin and modified starches. These ingredients, which are generally approved for food use, may give a faster release when used as an encapsulant for the physiological cooling agents. Other encapsulants like acacia or maltodextrin can also encapsulate the physiological cooling agents, but give a faster release rate of the physiological cooling agents.

5

10

15

20

25

30

The amount of coating or encapsulating material on the cooling agents also controls the length of time for their release from chewing gum.

Generally, the higher the level of coating and the lower the amount of active cooling agents, the slower the release of the cooling agents during mastication. To obtain the desired cooling agent release to blend with a gum's flavor release, the encapsulant should be a minimum of about 20% of the coated cooling agents. Preferably, the encapsulant should be a minimum of about 30% of the coated cooling agents, and most preferably should be a minimum of about 40% of the coated cooling agents. Depending on the coating material, a higher or lower amount of coating material may be needed to give the desired release of cooling agents.

Another method of giving a delayed release of the physiological cooling agents is agglomeration with an agglomerating agent which partially coats the cooling agents. This method includes the step of mixing the cooling agents and agglomerating agent with a small amount of water or solvent. The mixture is prepared in such a way as to have individual wet particles in contact with each other so a partial coating can be applied. After the water or solvent is removed, the mixture is ground and used as a powdered coated cooling agent.

Materials that can be used as the agglomerating agent are the same as those used in the encapsulation mentioned previously. However, since the coating is only a partial encapsulation, some agglomeration agents are

24

more effective in delaying release than others. Some of the better agglomerating agents are the organic polymers like acrylic polymer and copolymers, polyvinyl acetate, polyvinyl-pyrrolidone, waxes, shellac and Zein. Other agglomerating agents are not as effective in giving a delayed release as are the polymers, waxes, shellac and Zein, but can be used to give some delayed release. These others agglomerating agents include, but are not limited to, agar, alginates, a wide range of cellulose derivatives, dextrin, gelatin, modified starches, and vegetable gums like guar gums, locust bean gum, and carrageenan. Even though the agglomerated cooling agents are only partially coated, when the quantity of coating is increased compared to the quantity of the cooling agents, the release can be delayed for a longer time during mastication. The level of coating used in the agglomerated product is a minimum of about 5%. Preferably the coating level is a minimum of about 15%, and more preferably about 20%. Depending on the agglomerating agent, a higher or lower amount of agent may be needed to give the desired release of cooling agents.

5

10

15

20

25

30

The physiological cooling agents may be coated in a two-step process or multiple step process. The physiological cooling agents may be encapsulated with any of the materials as described previously and then the encapsulated material can be agglomerated as described previously to obtain an encapsulated/agglomerated product that could be used in chewing gum to give a delayed release.

In another embodiment of this invention, the physiological cooling agents may be absorbed onto another component, often referred to as a carrier, which is porous and become entrapped in the matrix of the porous component. Common materials used for absorbing the physiological cooling agents include, but are not limited to, silicas, silicates, pharmasorb clay, sponge-like beads or microbeads, amorphous carbonates and hydroxides, including aluminum and calcium lakes, vegetable gums and other spray dried materials.

Depending on the type of absorbent material and how it is prepared, the amount of the physiological cooling agents that can be loaded onto the absorbent will vary. Generally materials like polymers or spongelike beads or microbeads, amorphous sugars, and alditols and amorphous carbonates and hydroxides absorb about 10% to about 40% of the weight of the absorbent. Other materials like silicas and pharmasorb clays may be able to absorb about 20% to about 80% of the weight of the absorbent.

5

10

15

20

25

30

The general procedure for absorbing the physiological cooling agents onto the absorbent is as follows. An absorbent like fumed silica powder can be mixed in a powder blender and a solution of the physiological cooling agents can be sprayed onto the powder as mixing continues. The solution can be about 5% to 30% cooling agent, and higher levels may be used if higher temperatures are used. Generally water is the solvent, but other solvents like alcohol should also be used if approved for use in food. As the powder mixes, the liquid is sprayed onto the powder. Spraying is stopped before the mix becomes damp. The still flowing powder is removed from the mixer and dried to remove the water or other solvent, and ground to a specific particle size.

After the physiological cooling agents are absorbed onto an absorbent or fixed onto an absorbent, the fixative/cooling agents can be coated by encapsulation. Either full or partial encapsulation may be used, depending on the coating composition used in the process. Full encapsulation may be obtained by coating with a polymer as in spray drying, spray chilling, fluid-bed coating, extrusion, coacervation, or any other standard technique. A partial encapsulation or coating can be obtained by agglomeration of the fixative/cooling agents mixture using any of the materials discussed above.

The physiological cooling agents can be treated to modify their release by being entrapped in an extrusion process. Examples of such extrusion processes are disclosed in U.S. Patent No. 5,128,155 and PCT Publication No. WO 94/06308.

The four methods to use to obtain a modified release of physiological cooling agents are (1) encapsulation by spray drying, fluid-bed coating, spray chilling and coacervation to give full or partial encapsulation; (2) agglomeration to give partial encapsulation; (3) fixation or absorption which also gives partial encapsulation; and (4) entrapment by extrusion. These four methods, combined in any usable manner which physically isolates the physiological cooling agents, modifies their dissolvability or modifies the release of physiological cooling agents are included in this invention.

5

10

15

20

25

The previously described cooling flavor compositions and encapsulated, agglomerated or absorbed physiological cooling agents may readily be incorporated into a chewing gum composition. Generally the physiological cooling agents will be added to the gum in either the form of a cooling flavor composition or as part of a modified release combination of physiological cooling agents. However, both of these aspects of the invention may be used in the same gum formula, and the cooling flavor composition itself or its individual components may be treated to have a modified release. The remainder of the chewing gum ingredients are noncritical to the present invention. That is, the cooling flavor composition and/or coated particles of physiological cooling agents can be incorporated into conventional chewing gum formulations in a conventional manner. Naturally, the preferred chewing gum formulation is a sugarless formulation. However, the physiological cooling agents may also be used in a sugar chewing gum. The cooling flavor composition and coated physiological cooling agents may be used in either regular chewing gum or bubble gum.

In other embodiments of this invention, particularly coatings of chewing gum, the concentration of physiological cooling agent will depend on the intensity of the physiological cooling agent and the desired cooling effect. In general the concentration of cooling agents used is between about 0.001% and about 1% by weight of coating. The preferred concentration of cooling

10

15

20

25

30

agent is between about 0.01% and about 0.5%, more preferably between about 0.02% and about 0.2%.

The present invention contemplates that one or more flavors may be added to the syrup used to make the coating, or applied to the gum center while the syrup coating is drying, or after the coating has dried. Furthermore, the flavor may be applied anywhere within the sequence of coats, for example, after the third, twelfth, eighteenth, etc., coats.

These flavors include any flavor which is of food acceptable quality, including the flavors described earlier for use in chewing gum. The flavor may be added to the coating syrup in an amount such that the coating will contain from about 0.2% to about 1.2% flavor and preferably from about 0.7% to about 1.0% flavor.

For menthol flavored coatings, this concentration of physiological cooling agent allows a reduced overall concentration of menthol. However, it does not completely eliminate menthol, because menthol has a very identifiable unique taste and cooling sensation. Hence, in the case of menthol flavor, the physiological cooling agents only enhance the cooling while reducing the menthol bitterness.

Artificial sweeteners contemplated for use in the coating include but are not limited to synthetic substances, saccharin, thaumatin, alitame, saccharin salts, aspartame, sucralose and acesulfame-K. The artificial sweetener may be added to the coating syrup in an amount such that the coating will contain from about 0.05% to about 0.3%, and preferably from about 0.10% to about 0.15% artificial sweetener.

Dispersing agents are often added to syrup coatings for the purpose of whitening and tack reduction. Dispersing agents contemplated by the present invention to be employed in the coating syrup include titanium dioxide, talc, or any other antistick compound. Titanium dioxide is a presently preferred dispersing agent of the present invention. The dispersing agent may be added to the coating syrup in amounts such that the coating will

contain from about 0.1% to about 1.0%, and preferably from about 0.3% to about 0.6% of the agent.

5

10

15

20

25

Coloring agents are preferably added directly to the syrup in the dye or lake form. Coloring agents contemplated by the present invention include food quality dyes. Film formers preferably added to the syrup include methyl cellulose, gelatins, hydroxypropyl cellulose, ethyl cellulose, hydroxyethyl cellulose, carboxymethyl cellulose and the like and combinations thereof. Binding agents may be added either as an initial coating on the chewing gum center or may be added directly into the syrup. Binding agents contemplated by the present invention include gum arabic, alginate, cellulosics, vegetable gums and the like.

Conventional panning procedures generally use sucrose as the coating material, but recent advances in panning have allowed the use of other carbohydrate materials to be used in the place of sucrose. Some of these components include, but are not limited to, dextrose, maltose, erythritol, xylitol, hydrogenated isomaltulose, maltitol and other new polyols or a combination thereof.

The coating material may be blended with panning modifiers including, but not limited to, gum arabic, maltodextrins, corn syrup, gelatin, cellulose type materials like carboxymethyl cellulose or hydroxymethyl cellulose, starch and modified starches, vegetable gums like alginates, locust bean gum, guar gum and gum tragacanth, insoluble carbonates like calcium carbonate or magnesium carbonate, and talc. Antitack agents may also be added as panning modifiers, which allow the use of a variety of carbohydrates and sugar alcohols to be used in the development of new panned or coated gum products.

The chewing gum center of the present invention follows the general pattern outlined below. These centers may contain physiological cooling agents as an ingredient.

10

15

20

25

30

In general, a chewing gum center composition or other chewing gum compositions typically contain a chewable gum base portion which is essentially free of water and is water-insoluble, a water-soluble bulk portion and flavors which are typically water insoluble. The water-soluble portion dissipates with a portion of the flavor over a period of time during chewing. The gum base portion is retained in the mouth throughout the chew.

The insoluble gum base generally comprises elastomers, elastomer solvents, plasticizers, waxes, emulsifiers and inorganic fillers. Plastic polymers, such as polyvinyl acetate, which behave somewhat as plasticizers, are also often included. Other plastic polymers that may be used include polyvinyl laureate, polyvinyl alcohol and polyvinyl pyrrolidone.

Elastomers may include polyisobutylene, butyl rubber, (isobutylene-isoprene copolymer) and styrene butadiene rubber, as well as natural latexes such as chicle. Elastomer solvents are often resins such as terpene resins. Plasticizers, sometimes called softeners, are typically fats and oils, including tallow, hydrogenated and partially hydrogenated vegetable oils, and coca butter. Commonly employed waxes include paraffin, microcrystalline and natural waxes such as beeswax and carnauba. Microcrystalline waxes, especially those with a high degree of crystallinity, may be considered bodying agents or textural modifiers.

According to the preferred embodiment of the present invention, the insoluble gum base constitutes between about 5% to about 95% by weight of the gum. More preferably the insoluble gum base comprises between 10% and 50% by weight of the gum and most preferably about 20% to 35% by weight of the gum.

The gum base typically also includes a filler component. The filler component may be calcium carbonate, magnesium carbonate, talc, dicalcium phosphate or the like. The filler may constitute between about 5% and about 60% by weight of the gum base. Preferably the filler comprises about 5% to 50% by weight of the gum base.

Gum bases typically also contain softeners including glycerol monostearate and glycerol triacetate. Gum bases may also contain optional ingredients such as antioxidants, colors, and emulsifiers. The present invention contemplates employing any commercially acceptable gum base.

5

The water-soluble portion of the chewing gum may further comprise softeners, sweeteners, flavors, physiological cooling agents and combinations thereof. The sweeteners often fulfill the role of bulking agents in the gum. The bulking agents typically comprise about 5% to about 95% of the gum composition.

10

Softeners are added to the chewing gum in order to optimize the chewability and mouth feel of the gum. Softeners, also known in the art as plasticizers or plasticizing agents, generally constitute between about 0.5% to about 15% of the chewing gum. Softeners contemplated by the present invention include glycerin, lecithin and combinations thereof. Further, aqueous sweetener solutions such as those containing sorbitol, hydrogenated starch hydrolysate, corn syrup and combinations thereof may be used as softeners and binding agents in gum.

20

15

As mentioned above, the cooling flavor compositions or coated physiological cooling agents of the present invention will most likely be used in sugarless gum formulations. However, formulations containing sugar are also within the scope of the invention. Sugar sweeteners generally include saccharide-containing components commonly known in the chewing gum art which comprise, but are not limited to, sucrose, dextrose, maltose, dextrin, dried invert sugar, fructose, galactose, corn syrup solids and the like, alone or in any combination.

25

30

The cooling flavor compositions and coated physiological cooling agents of the present invention can also be used in combination with sugarless sweeteners. Generally sugarless sweeteners include components with sweetening characteristics but which are devoid of the commonly known sugars and comprise, but are not limited to, sugar alcohols such as sorbitol,

10

15

20

25

30

hydrogenated isomaltulose, mannitol, xylitol, lactitol, erythintol, hydrogenated starch hydrolysate, maltitol and the like alone or in any combination.

Depending on the particular sweetness release profile and shelf-stability needed, coated or uncoated high-intensity sweeteners may be used in the chewing gum center or in the coating. High-intensity sweeteners, preferably aspartame, may be used at levels from about 0.01% to about 3.0%. Encapsulated aspartame is a high intensity sweetener with improved stability and release characteristics, as compared to free aspartame. Free aspartame can also be added, and a combination of some free and encapsulated aspartame is preferred when aspartame is used.

Optional ingredients such as colors, emulsifiers and pharmaceutical agents may also be added as separate components of the chewing gum composition, or added as part of the gum base.

Aqueous syrups, such as corn syrup and hydrogenated corn syrup may be used, particularly if their moisture content is reduced. This can preferably be done by coevaporating the aqueous syrup with a plasticizer, such as glycerin or propylene glycol, to a moisture content of less than 10%. Preferred compositions include hydrogenated starch hydrolysate solids and glycerin. Such syrups and their methods of preparation are discussed in detail in U.S. Patent No. 4,671,967.

A preferred method of manufacturing chewing gum according to the present invention is by sequentially adding the various chewing gum ingredients to any commercially available mixer known in the art. After the ingredients have been thoroughly mixed, the gum is discharged from the mixer and shaped into the desired form such as by rolling into sheets and cutting into sticks, extruding into chunks, or casting into pellets.

Generally, the ingredients are mixed by first melting the gum base and adding it to the running mixer. the base may also be melted in the mixer itself. Color or emulsifiers may also be added at this time, along with syrup and a portion of the bulking agent. Further portions of the bulking agent may

10

15

20

25

30

then be added to the mixer. A flavoring agent is typically added with the final portion of the bulking agent. The cooling flavor composition of the present invention are preferably added as part of the flavor addition. The coated physiological cooling agents of the present invention are preferably added after the final portion of bulking agent and flavor have been added. The entire mixing procedure typically takes from five to fifteen minutes, but longer mixing times may sometime be required. Those skilled in the art will recognize that many variations of the above described procedures may be followed.

When the physiological cooling agent is used as part of a cooling flavor composition, using menthol and menthone, the cooling flavor composition will contain preferably about 40% to about 80%, and more preferably about 50% to about 70%, menthol, and about 15% to about 50%, and more preferably about 20% to about 40%, menthone. In the preferred cooling flavor composition, the ratio of menthol to menthone is preferably between about 4:5 and about 16:3. The ratio of physiological cooling agents to the total of menthol and menthone is preferable between about 3:97 and about 1:4. Typically this will mean about 3% to about 25%, and more preferably about 4% to about 15% of the cooling composition will be one or more physiological cooling agents. The preferred physiological cooling agents are: menthyl succinate; N-2,3-trimethyl-2-isopropyl butanamide; menthyl lactate; 3-l-menthoxypropane-1,2-diol; N-substituted p-menthane carboxamide; menthone glycerol ketals and mixture thereof.

If another flavor is used in the gum, or other oral composition, beside the cooling flavor composition, the cooling flavor composition outlined above may be mixed with the other flavor. In addition, some components of the cooling flavor composition may be mixed with the other flavor, and some components added separately to the gum. However, it is preferred that any other flavor and the cooling flavor composition be premixed before being added to the gum.

PCT/US97/16731

WO 99/13734

5

10

15

20

25

30

Once formed, the chewing gum center can be coated. The coating is initially present as a liquid syrup which contains from about 30% to about 80% or 85% of the coating ingredients previously described herein, and from about 15% or 20% to about 70% of a solvent such as water. In general, the coating process is carried out in conventional panning equipment. Sugarless gum center tablets to be coated are placed into the panning equipment to form a moving mass.

The material or syrup which will eventually form the coating is applied or distributed over the gum center tablets. Flavors may be added before, during and after applying the syrup to the gum centers. Once the coating has dried to form a hard surface, additional syrup additions can be made to produce a plurality of coatings or multiple layers of coating.

In the panning procedure, syrup is added to the gum center tablets at a temperature range of from about 100°F. to about 240°F. Preferably, the syrup temperature is from about 140°F. to about 200°F. Most preferably, the syrup temperature should be kept constant throughout the process in order to prevent the polyol in the syrup from crystallizing. The syrup may be mixed with, sprayed upon, poured over, or added to the gum center tablets in any way known to those skilled in the art.

In another embodiment, a soft coating is formed by adding a powder coating after a liquid coating. The powder coating may include natural carbohydrate gum hydrolysates, maltodextrin, gelatin, cellulose derivatives, starches, modified starches, sugars, sugar alcohols, natural carbohydrate gums and fillers like talc and calcium carbonate.

Each component of the coating on the gum center may be applied in a single layer or in a plurality of layers. In general, a plurality of layers is obtained by applying single coats, allowing the layers to dry, and then repeating the process. The amount of solids added by each coating step depends chiefly on the concentration of the coating syrup. Any number of coats may be applied to the gum center Tablet. Preferably, no more than

about 75 coats are applied to the gum center. More preferably, less than about 60 coats are applied and most preferably, about 30 to about 60 coats are applied. In any event, the present invention contemplates applying an amount of syrup sufficient to yield a coated chewing gum product containing about 10% to about 65% coating. Preferably, the final product will contain from about 20% to about 50% coating.

5

10

15

20

25

Those skilled in the art will recognize that in order to obtain a plurality of coated layers, a plurality of premeasured aliquots of coating syrup may be applied to the gum center. It is contemplated, however, that the volume of aliquots of syrup applied to the gum center may vary throughout the coating procedure.

Once a coating of syrup is applied to the gum center, the present invention contemplates drying the wet syrup in an inert medium. A preferred drying medium comprises air. Preferably, forced drying air contacts the wet syrup coating in a temperature range of from about 70°F to about 110°F. More preferably, the drying air is in the temperature range of from about 80°F to about 100°F. The invention also contemplates that the drying air possess a relative humidity of less than about 15 percent. Preferably, the relative humidity of the drying air is less than about 8 percent.

The drying air may be passed over and admixed with the syrup coated gum centers in any way commonly known in the art. Preferably, the drying air is blown over and around the syrup coated gum center at a flow rate, for large scale operations, of about 2800 cubic feet per minute. If lower quantities of material are being processed, or if smaller equipment is used, lower flow rates would be used. If a flavor is applied after a syrup coating has been dried, the present invention contemplates drying the flavor with or without the use of a drying medium.

#### **EXAMPLES**

The following four ingredients can be mixed with flavors and used in chewing gum formulations. The percentage listed are percentages of the total of these four ingredients. However, the ingredients were not mixed together first, but were added individually to the flavor used in the gum. The four ingredients in combination are referred to herein as cooling blends A, B and C.

	Α	В	С
Menthol	52.5	60.2	62.5
Menthone	25.8	30.6	31.2
Menthyl succinate	14.5	6.1	4.2
Menthyl lactate	7.2	3.1	2.1

## Examples 1 and 2

Cooling blend A may be mixed with a cherry flavor and used to make
two cherry flavored sugarless gum formulas. Both products would have a
slightly minty flavor with cherry. When a peppermint flavor is used in place of
the cooling blend in comparable formulas, the minty character is too strong.
The cooling blend gives a high quality cool cherry mint flavored product as
formulated below:

TABLE 1

V			
	Example 1	Example 2	_
Sorbitol	49.51	51.44	
Base	28.50	25.00	
Glycerin	12.00	12.00	
Cherry Flavor	3.0848	2.8848	
Cooling Blend A	0.1152	0.1152	
Spray Dried Cherry Flavor	0.30		
Xylitol	1.50	4.50	
Fumeric, Citric, and Adipic Acids	2.10	2.10	
Encapsulated Sweeteners	1.36	1.36	
Salt Solution	0.75	_	
Color	0.28	0.30	
Lecithin	0.50	0.30	
TOTAL	100.00	100.00	

WO 99/13734

# Examples 3, 4 and 5

Cooling blend B may be used in a sugarless wintergreen flavored chewing gum to give improved cooling without mint flavor according to the following formulas:

	TABLE 2		
	Example 3	Example 4	Example 5
Sorbitol	49.60	50.01	48.76
Base	25.10	25.10	25.10
Glycerin	14.00	6.00	14.00
Maltitol	5.00	7.00	10.00
Coevaporated Glycerin/Lycasin*	3.50	9.50	
Wintergreen Flavor	1.1296	1.1296	1.1296
Cooling Blend B	0.4704	0.4704	0.4704
Encapsulated Wintergreen Flavor	0.1625	0.1625	
Encapsulated Menthol	0.0875	0.0875	_
Encapsulated Sweeteners	0.54	0.54	0.54
Salt Solution**	0.28		
Color	0.13		_
TOTAL	100.00	100.00	100.00

<sup>\*</sup> Contains 25% glycerin, 67.5% Lycasin brand hydrogenated starch hydrolsate solids and 7.5% water.

<sup>\*\*</sup> Contains 10% NaCl and 90% water.

# Examples 6 through 10

Cooling blend C was used in spearmint and peppermint flavored chewing gums of Examples 6 through 10 to give improved cooling with mint flavor. The increased cooling yielded products with less bitterness and harshness, and gave good minty tasting products. The following formulas were made:

TABLE 3

	Example 6	Example 7	Example 8	Example 9	Example 10
Sorbitol	47.21	48.60	32.76	48.41	40.60
Base	25.00	25.00	37.35	25.00	30.00
Glycerin	6.00	11.40	12.00	8.00	10.00
Coevaporated Glycerin/Lycasin*	9.50	7.67	_	9.50	3.50
Maltitol <sup>A</sup> /Mannitol <sup>B</sup>	10.00 <sup>A</sup>	5.00 <sup>A</sup>	3.07 <sup>B</sup>	5.00 <sup>8</sup>	7.00 <sup>B</sup>
Spearmint Flavor	1.0908	1.0908			
Cooling Blend C	0.2592	0.2592	0.7425	0.7425	0.40
Peppermint Flavor		_	1.5075	1.5075	1.60
Lecithin	0.30	0.30	0.40	0.30	0.30
Calcium Carbonate	_		9.66	_	5.00
Encapsulated Menthol	_	0.20	1.50	1.00	1.00
Encapsulated Sweetener	0.54	0.34	0.34	0.54	- 0.60
Salt Solution**	0.10	0.10	0.67		
Color	_	0.04	_	_	_
TOTAL	100.00	100.00	100.00	100.00	100.00

<sup>\*</sup> Contains 25% glycerin, 67.5% Lycasin brand hydrogenated starch hydrolsate solids and 7.5% water.

<sup>\*\*</sup> Contains 10% NaCl and 90% water.

# **Examples 11 through 17**

The following formulations contain combinations of cooling agents that may give fast and slow release and may be synergistic to increase cooling when used in the same formulation:

TABLE 4
SUGARLESS CHEWING GUM

	Example 11	Example 12	Example 13	Example 14
Base	33.10	28.00	30.00	28.00
Calcium Carbonate	_	_	11.00	<del></del>
Sorbitol	55.90	54.36	46.08	49.52
Glycerin	8.00	5.40	0.40	8.30
Liquid Sorbitol	_	6.00	6.20	2.00
Encapsulated Sweetener	0.70	0.24	0.24	0.82
Menthol	1.10	_	0.26	0.30
Active ML/TCA*	0.10	0.20	0.12	0.16
Mannitol		3.00	3.00	8.00
Peppermint Flavor	1.10	2.00		
Methyl Salicylate Flavor		_	1.90	1.90
Lecithin	_	0.80	0.80	1.00
Encapsulated Menthol/Flavor		_		
TOTAL	100.00	100.00	100.00	100.00

<sup>\* 50/50</sup> combination of ML and TCA

TABLE 5
SUGARLESS CHEWING GUM

	Example 15	Example 16	Example 17
Base	29.00	31.60	33.60
Calcium Carbonate	15.00	11.00	13.00
Sorbitol	41.26	44.48	47.73
Glycerin	10.00	0.80	0.75
Liquid Sorbitol	_	7.20	_
Encapsulated Sweetener	0.40	0.66	0.66
Menthoi	0.90	0.30	0.30
Active ML/MGK *	0.14	0.06	0.06
Mannitol			
Peppermint Flavor	1.30	1.40	1.40
Methyl Salicylate Flavor	_		
Lecithin	_	0.50	0.50
Encapsulated Menthol/Flavor	2.00	2.00	2.00
TOTAL	100.00	100.00	100.00

<sup>\* 50/50</sup> combination of ML and MGK

N-substituted p-menthane carboxamides, for example N-ethyl-p-menthane-3-carboxamide (WS-3), when combined with menthone ketals such as menthone glycerol ketal, may produce synergistic cooling effects in chewing gum. It is also possible to take advantage of differing release rates of the two cooling agents to provide a fast cooling release and a long lasting cooling in the same product. For example, chewing gums may be prepared form the following formulas:

**TABLE 6** 

	Example 18	Example 19
Gum Base	25.00	30.00
Sugar	57.11	58.46
Corn Syrup	15.00	9.00
Glycerin	2.00	1.50
Spearmint Oil	0.70	
Peppermint Oil		0.90
WS-3	0.08	0.06
Menthone Glycerol Ketal	<u>0.11</u>	0.08
TOTAL	100.00	100.00

10

Additional cooling agents may also be added as in some of the following examples:

TABLE 7

	Example 20	Example 21	Example 22	Example 23	Example 24	Example 25
Gum Base	48.00	35.00	30.00	25.00	22.00	30.00
Sorbitol	34.05	49.05	53.07	60.69	59.54	51.12
Hydrogenated Starch	_	_	_	7.5	14.00	10.00
Hydrolysate Syrup						
70% Sorbitol Solution	10.00	8.00		_		
Glycerin	5.00	6.00	15.00	5.00	3.00	7.00
Encapsulated Aspartame	0.30	0.50	0.60	0.20	0.30	0.40
Methyl Salicylate	0.50	_	_	-	0.70	
Peppermint Oil	1.30	0.40	_	0.95	0.20	1.20
Spearmint Oil	0.20	0.80	0.80	_	_	
Ŵ\$-3	0.30	0.10	0.08	0.11	0.13	0.04
Menthane Glycerol Ketal	0.05	0.10	0.20	0.35	0.05	0.04
WS-23		0.05	0.05		_	_
Menthyl Lactate	0.30	_	0.20		_	0.10
Menthyl Succinate	_			0.20	0.08	0.10
•	100.00	100.00	100.00	100.00	100.00	100.00

Acyclic carboxamides, for example N-2,3-trimethyl-2-isopropyl butanamide (WS-23), may produce synergistic cooling effects when combined with menthone ketals, such as menthone glycerol ketal, in chewing gum. It is also possible to take advantage of differing release rates of the two cooling agents to provide a fast cooling release and a long lasting cooling in the same product. Also, reductions in bitterness may be achieved through optimization of the levels of these cooling agents. For example, chewing gums may be prepared from the following formulas:

TABLE 8

Example 27
30.00%
58.46%
9.00%
1.50%
0.90%
0.06%
0.08%
100.00

Additional cooling agents may also be added as in some of the following examples:

TABLE 9

	Example 28	Example 29	Example 30	Example 31	Example 32	Example 33
Gum Base	48.00	35.00	30.00	25.00	22.00	30.00
Sorbitol	34.05	48.90	53.07	60.69	59.54	50.87
Hydrogenated Starch Hydrolysate Syrup	_	-	_	7.50	14.00	10.00
70% Sorbitol Solution	10.00	8.00	_		_	
Glycerin	5.00	6.00	15.00	5.00	3.00	7.00
Encapsulated Aspartame	0.30	0.50	0.60	0.20	0.30	0.40
Methyl Salicylate	0.50	_	_		0.70	
Peppermint Oil	1.30	0.40	_	0.95	0.20	1.20
Spearmint Oil	0.20	0.80	0.80	<del>-</del> .		_
WS-23	0.30	0.10	0.08	0.11	0.13	0.04
Menthone Glycerol Ketal	0.05	0.10	0.20	0.35	0.05	0.04
WS-3	_	0.05	0.05	_		
Menthyl Lactate	0.30		0.20	_	_	0.10
Menthyl Succinate		_	_	0.20	. 0.08	0.10
Menthol		0,15		_		0.25
	100.00	100.00	100.00	100.00	100.00	100.00

Acyclic carboxamides, for example N-2,3-trimethyl-2-isopropyl

butanamide (WS-23), may produce synergistic cooling effects when
combined with menthol in chewing gum. It is also possible to take advantage
of differing release rates of the two cooling agents to provide a fast cooling
release and a long lasting cooling in the same product. Also, reductions in
bitterness may be achieved through optimization of the levels of these
cooling agents. For example, chewing gums may be prepared from the
following formulas:

TABLE 10

	Example 34	Example 35
Gum Base	25.00%	30.00%
Sugar	56. <b>76</b> %	58.29%
Corn Syrup	15.00%	9.00%
Glycerin	2.00%	1.50%
Spearmint Oil	0.70%	_
Peppermint Oil	<del></del>	0.90%
WS-23	0.04%	0.06%
Menthol	0. <u>50%</u>	0.25%
TOTAL	100.00	100.00

10

Additional cooling agents may also be added as in some of the following examples:

TABLE 11

	Example 36	Example 37	Example 38	Example 39	Example 40	Example 41
Gum Base	48.00	35.00	30.00	25.00	22.00	30.00
Sorbitol	33.85	48.60	53.12	60.69	59.54	50.46
Hydrogenated Starch Hydrolysate Syrup	_	_	_	7.50	14.00	10.00
70% Sorbitol Solution	10.00	8.00	_	_	_	_
Glycerin	5.00	6.00	15.00	5.00	3.00	7.00
Encapsulated Aspartame	0.30	0.50	0.60	0.20	0.30	0.40
Methyl Salicylate	0.50	_	_	_	0.70	_
Peppermint Oil	1.30	0.40	_	0.95	0.20	1.20
Spearmint Oil	0.20	0.80	0.80			_
WS-23	0.30	0.10	0.08	0.11	0.13	0.04
Mentho!	0.25	0.40	0.15	0.35	0.05	0.45
WS-3	_	0.05	0.05			
Menthyl Lactate	0.30		0.20	_		0.10
Menthyl Succinate	_	_		0.20	0.08	0.10
Menthone Glycerol Ketal		0.15				0.25
	100.00	100.00	100.00	100.00	100.00	100.00

Menthyl succinate may produce synergistic cooling effects when combined with acyclic carboxamides, for example N-2,3-trimethyl-2-isopropyl butanamide (WS-23), in chewing gum. It is also possible to take advantage of differing release rates of the two cooling agents to provide a fast cooling release and a long lasting cooling in the same product. Also, reductions in bitterness may be achieved through optimization of the levels of these cooling agents. For example, chewing gums may be prepared from the following formulas:

TABLE 12

	Example 42	Example 43
Gum Base	25.00%	30.00%
Sugar	57.11%	58.46%
Com Syrup	15.00%	9.00%
Glycerin	2.00%	1.50%
Spearmint Oil	0.70%	-
Peppermint Oil	<del></del>	0.90%
WS-23	0.08%	0.06%
Menthyl Succinate	0.11%	_0.08%
TOTAL	100.00	100.00

10

Additional cooling agents may also be added as in some of the following examples:

TABLE 13

	Example 44	Example 45	Example 46	Example 47	Example 48	Example 49
Gum Base	48.00	35.00	30.00	25.00	22.00	30.00
Sorbitol	34.05	48.85	52.57	60.69	59.24	51.12
Hydrogenated Starch Hydrolysate Syrup	. —		-	7.50	14.00	10.00
70% Sorbitol Solution	10.00	8.00	_	_	_	_
Glycerin	5.00	6.00	15.00	5.00	3.00	7.00
Encapsulated Aspartame	0.30	0.50	0.60	0.20	0.30	0.40
Methyl Salicylate	0.50		_		<b>0</b> .70	_
Peppermint Oil	1.30	0.40	_	0.95	0.20	1.20
Spearmint Oil	0.20	0.80	0.80		-	
ŴS-3			0.08	0.16	_	_
Menthone Glycerol Ketal		· —	_	-	0.05	0.04
WS-23	0.30	0.05	0.05	0.20	0.13	0.04
Menthyl Lactate	0.30	0.10	0.20	0.10	-	0.10
Menthyl Succinate	0.05	0.10	0.20	0.20	0.08	0.10
Menthol		0.20	0.50		0.30	
	100.00	100.00	100.00	100.00	100.00	100.00

Menthyl succinate may produce synergistic cooling effects when combined with menthone ketals, such menthone glycerol ketal, in chewing gum. It is also possible to take advantage of differing release rates of the two cooling agents to provide a fast cooling release and a long lasting cooling in the same product. Also, reductions in bitterness may be achieved through optimization of the levels of these cooling agents. For example, chewing gums may be prepared from the following formulas:

TABLE 14

	Example 50	Example 51
Gum Base	25.00%	30.00%
Sugar	57.11%	58.46%
Com Syrup	15.00%	9.00%
Glycerin	2.00%	1.50%
Spearmint Oil	0.70%	
Peppermint Oil	<del>-</del>	0.90%
Menthyl Succinate	0.08%	0.06%
Menthone Glycerol Ketal	0.11%	0.08%
TOTAL	100.00	100.00

10

WO 99/13734 PCT/US97/16731

44

Additional cooling agents may also be added as in some of the following examples:

TABLE 15

	Example 52	Example 53	Example 54	Example 55	Example 56	Example 57
Gum Base	48.00	35.00	30.00	25.00	22.00	30.00
Sorbitol	34.05	48.85	52.57	60.69	59.24	51.12
Hydrogenated Starch Hydrolysate Syrup	_			7.50	14.00	10.00
70% Sorbitol Solution	10.00	8.00	_	_		_
Glycerin	5.00	6.00	15.00	5.00	3.00	7.00
Encapsulated Aspartame	0.30	0.50	0.60	0.20	0.30	0.40
Methyl Salicylate	0.50	_		_	0.70	
Peppermint Oil	1.30	0.40	_	0.95	0.20	1.20
Spearmint Oil	0.20	0.80	0.80	_	-	_
WS-3	_			0.05	0.08	
Menthone Glycerol Ketal	0.05	0.10	0.20	0.35	0.10	0.07
WS-23	_	0.05	0.05		_	
Menthyl Lactate	0.30	_	0.20		_	0.10
Menthyl Succinate	0.30	0.10	0.08	0.26	0.08	0.11
Menthol		0.20	0.50		0.30	
	100.00	100.00	100.00	100.00	100.00	100.00

Menthyl succinate may produce synergistic cooling effects when combined with menthyl lactate in chewing gum. It is also possible to take advantage of differing release rates of the two cooling agents to provide a fast cooling release and a long lasting cooling in the same product. Also, reductions in bitterness may be achieved through optimization of the levels of these cooling agents. For example, chewing gums may be prepared from the following formulas:

TABLE 16

	Example 58	Example 59
Gum Base	25.00%	30.00%
Sugar	57.11%	58.46%
Corn Syrup	15.00%	9.00%
Glycerin	2.00%	1.50%
Spearmint Oil	0.70%	<del>-</del>
Peppermint Oil	_	0.90%
Menthyl Lactate	0.08%	0.06%
Menthyl Succinate	0.11%	0.08%
TOTAL	100.00	100.00

10

Additional cooling agents may also be added as in some of the following examples:

TABLE 17

,	Example 60	Example 61	Example 62	Example 63	Example 64	Example 65
Gum Base	48.00	35.00	30.00	25.00	22.00	30.00
Sorbitol	34.05	48.85	52.57	60.69	59.24	51.12
Hydrogenated Starch	. <b>–</b>	_	_	7.50	14.00	10.00
Hydrolysate Syrup						
70% Sorbitol Solution	10.00	8.00	_	_		_
Glycerin	5.00	6.00	15.00	5.00	3.00	7.00
Encapsulated Aspartame	0.30	0.50	0.60	0.20	0.30	0.40
Methyl Salicylate	0.50		_		0.70	_
Peppermint Oil	1.30	0.40	_	0.95	0.20	1.20
Spearmint Oil	0.20	0.80	0.80			_
WS-3	0.10	_	0.08	_		0.04
Menthone Glycerol Ketal	0.05	_		0.11	0.05	0.04
WS-23		0.05	0.05			_
Menthyl Lactate	0.30	0.10	0.25	0.35	0.13	0.10
Menthyl Succinate	0.20	0.10	0.15	0.20	0.08	0.10
Menthol		0.20	0.50		0.30	
	100.00	100.00	100.00	100.00	100.00	100.00

Menthyl succinate may produce synergistic cooling effects when combined with N-substituted p-menthane carboxamides, for example N-ethyl-p-menthane-3-carboxamide (WS-3), in chewing gum. It is also possible to take advantage of differing release rates of the two cooling agents to provide a fast cooling release and a long lasting cooling in the same product. Also, reductions in bitterness may be achieved through optimization of the levels of these cooling agents. For example, chewing gums may be prepared from the following formulas:

TABLE 18

	Example 66	Example 67
Gum Base	25.00%	30.00%
Sugar	57.11%	58.46%
Com Syrup	15.00%	9.00%
Glycerin	2.00%	1.50%
Spearmint Oil	0.70%	
Peppermint Oil	<del>-</del>	0.90%
WS-3	0.08%	0.06%
Menthyl Succinate	0.11%	0.08%
TOTAL	100.00	100.00

WO 99/13734

5

10

Additional cooling agents may also be added as in some of the following examples:

TABLE 19

	Example 68	Example 69	Example 70	Example 71	Example 72	Example 73
Gum Base	48.00	35.00	30.00	25.00	22.00	30.00
Sorbitol	34.05	48.85	52.57	60.69	59.24	51.12
Hydrogenated Starch Hydrolysate Syrup	_	_	_	7.50	14.00	10.00
70% Sorbitol Solution	10.00	8.00	_			_
Glycerin	5.00	6.00	15.00	5.00	3.00	7.00
Encapsulated Aspartame	0.30	0.50	0.60	0.20	0.30	0.40
Methyl Salicylate	0.50			_	0.70	_
Peppermint Oil	1.30	0.40	_	0.95	0.20	1.20
Spearmint Oil	0.20	0.80	0.80	_	_	
WS-3	0.30	0.10	0.08	0.25	0.13	0.04
Menthone Glycerol Ketal		_	_	0.05	0.05	0.04
WS-23		0.05	0.05	0.06		
Menthyl Lactate	0.30		0.20	0.10	_	0.10
Menthyl Succinate	0.05	0.10	0.20	0.20	0.08	0.10
Menthol		0.20	0.50		0.30	
	100.00	100.00	100.00	100.00	100.00	100.00

N-substituted p-menthane carboxamides, for example N-ethyl-p-menthane-3-carboxamide (WS-3), may produce synergistic cooling effects when combined with acyclic carboxamides, for example N-2,3-trimethyl-2-isopropyl-butanamide (WS-23), in chewing gum. It is also possible to take advantage of differing release rates of the two cooling agents to provide a fast cooling release and a long lasting cooling in the same product. Also, reductions in bitterness may be achieved through optimization of the levels of these cooling agents. For example, chewing gums may be prepared from the following formulas:

TABLE 20

	Example 74	Example 75
Gum Base	25.00%	30.00%
Sugar	57.11%	58.46%
Corn Syrup	15.00%	9.00%
Glycerin	2.00%	1.50%
Spearmint Oil	0.70%	_
Peppermint Oil	<del></del>	0.90%
WS-3	0.08%	0.06%
WS-23	_0.11%	0.08%
TOTAL	100.00	100.00

10

Additional cooling agents may also be added as in some of the following examples:

TABLE 21

	Example 76	Example 77	Example 78	Example 79	Example 80	Example 81
Gum Base	48.00	35.00	30.00	25.00	22.00	30.00
Sorbitol	34.05	48.85	52.57	60.69	59.24	51.08
Hydrogenated Starch	_	_	_	7.50	14.00	10.00
Hydrolysate Syrup						
70% Sorbitol Solution	10.00	8.00	_		_	_
Glycerin	5.00	6.00	15.00	5.00	3.00	7.00
Encapsulated Aspartame	0.30	0.50	0.60	0.20	0.30	0.40
Methyl Salicylate	0.50		_		0.70	_
Peppermint Oil	1.30	0.40		0.95	0.20	1.20
Spearmint Oil	0,20	0.80	0.80	_		_
WS-3	0.30	0.10	0.08	0.21	0.13	0.04
Menthone Glycerol Ketal	_		_	0.10	0.05	0.04
WS-23	0.05	0.15	0.25	0.10	0.04	0.10
Menthyl Lactate	<b>0</b> .30	_	0.20	0.05		0.07
Menthyl Succinate	_			0.20	0.04	0.07
Menthol	_	0.20	0.50	_	0.30	
	100.00	100.00	100.00	100.00	100.00	100.00

Menthyl salicylate (wintergreen) may produce synergistic cooling effects when combined with acyclic carboxamides, for example N-2,3-trimethyl-2-isopropyl butanamide (WS-23), in chewing gum. It is also possible to take advantage of differing release rates of the two cooling agents to provide a fast cooling release and a long lasting cooling in the same product. Also, reductions in bitterness may be achieved through optimization of the levels of these cooling agents. For example, chewing gums may be prepared from the following formulas:

TABLE 22

	Example 82	Example 83
Gum Base	25.00%	30.00%
Sugar	57.11%	58.46%
Com Syrup	15.00%	9.00%
Glycerin	2.00%	1.50%
Spearmint Oil	0.70%	_
Peppermint Oil	<del>-</del>	0.90%
WS-23	0.08%	0.06%
Methyl salicylate	0.11%	0.08%
TOTAL	100.00	100.00

10

Additional cooling agents may also be added as in some of the following examples:

TABLE 23

	Example 84	Example 85	Example 86	Example 87	Example 88	Example 89
Gum Base	48.00	35.00	30.00	25.00	22.00	30.00
Sorbitol	34.05	48.40	51.77	60.74	59.24	51.02
Hydrogenated Starch Hydrolysate Syrup	_	_		7.50	14.00	10.00
70% Sorbitol Solution	10.00	8.00		_	_	_
Glycerin	5.00	6.00	15.00	5.00	3.00	7.00
Encapsulated Aspartame	0.30	0.50	0.60	0.20	0.30	0.40
Methyl Salicylate	. 0.70	0.80	1.20	0.70	0.90	1.00
Peppermint Oil	1.00	0.40		0.20	_	
Spearmint Oil	0.10		0.40		_	0.30
WS-3	0.20	0.50	0.08		_	0.04
Menthone Glycerol Ketal	0.05	0.10	0.20	0.35	0.05	0.04
WS-23	0.30	0.10	0.05	0.11	0.13	0.08
Menthyl Lactate	0.30	_	0.20	_	<del></del>	0.06
Menthyl Succinate		_	_	0.20	0.08	0.06
Menthol		0.20	0.50		0.30	
	100.00	100.00	100.00	100.00	100.00	100.00

Menthyl salicylate (wintergreen) may produce synergistic cooling effects when combined with N-substituted p-menthane carboxamides, for example N-ethyl-p-menthane-3-carboxamide (WS-3), in chewing gum. It is also possible to take advantage of differing release rates of the two cooling agents to provide a fast cooling release and a long lasting cooling in the same product. Also, reductions in bitterness may be achieved through optimization of the levels of these cooling agents. For example, chewing gums may be prepared from the following formulas:

TABLE 24

	Example 90	Example 91
Gum Base	25.00%	30.00%
Sugar	57.11%	58,46%
Com Syrup	15.00%	9.00%
Glycerin	2.00%	1.50%
Spearmint Oil	0.70%	<u> </u>
Peppermint Oil		0.90%
WS-3	0.08%	0.06%
Methyl Salicylate	0.11%	0.08%
TOTAL	100.00	100.00

10

Additional cooling agents may also be added as in some of the following examples:

TABLE 25

	Example 92	Example 93	Example 94	Example 95	Example 96	Example 97
Gum Base	48.00	35.00	30.00	25.00	22.00	30.00
Sorbitol	34.05	48.85	51.77	59.75	58.94	51.02
Hydrogenated Starch	_	_	_	7.50	14.00	10.00
Hydrolysate Syrup						
70% Sorbitol Solution	10.00	8.00				
Glycerin	5.00	6.00	15.00	5.00	3.00	7.00
Encapsulated Aspartame	0.30	0.50	0.60	0.20	0.30	0.40
Methyl Salicylate	0.85	0.80	1.20	0.70	0.90	1.00
Peppermint Oil	1.00	0.40	_	1.20	0.20	_
Spearmint Oil	0.15	_	0.40	_		0.30
WS-3	0.25	0.20	0.08	0.20	0.13	0.04
Menthone Glycerol Ketal			0.10	0.25	0.05	0.04
WS-23	0.10	0.05	0.05	_	0.10	
Menthyl Lactate	0.30	_	0.20	_		0.10
Menthyl Succinate	_	_	0.10	0.20	0.08	0.10
Menthol		0.20	0.50		0.30	
	100.00	100.00	100.00	100.00	100.00	100.00

The cooling agent 3-l-menthoxypropane-1,2-diol (TCA) from Takasago may produce synergistic cooling effects when combined with menthone ketals, such as menthone glycerol ketal, in chewing gum. It is also possible to take advantage of differing release rates of the two cooling agents to provide a fast cooling release and a long lasting cooling in the same product. Also, reductions in bitterness may be achieved through optimization of the levels of these cooling agents. For example, chewing gums may be prepared from the following formulas:

TABLE 26

	Example 98	Example 99
Gum Base	25.00%	30.00%
Sugar	<b>57.11%</b>	58.46%
Com Syrup	15.00%	9.00%
Glycerin	2.00%	1.50%
Spearmint Oil	0.70%	_
Peppermint Oil		0.90%
TCA	0.08%	0.06%
Menthone Glycerol Ketal	<u>0.11%</u>	0.08%
TOTAL	100.00	100.00

10

Additional cooling agents may also be added as in some of the following examples:

TABLE 27

	Example 100	Example 101	Example 102	Example 103	Example 104	Example 105
Gum Base	48.00	35.00	30.00	25.00	22.00	30.00
Sorbitol	34.05	48.90	53.07	60.69	59.54	50.87
Hydrogenated Starch Hydrolysate Syrup	-		_	7.50	14.00	10.00
70% Sorbitol Solution	10.00	8.00			_	
Glycerin	5.00	6.00	15.00	5.00	3.00	7.00
Encapsulated Aspartame	0.30	0.50	0.60	0.20	0.30	0.40
Methyl Salicylate	0.50			_	0.70	_
Peppermint Oil	1.30	0.40		0.95	0.20	1.20
Spearmint Oil	0.20	0.80	0.80	_		_
TCA	0.30	0.10	0.08	0.11	0.13	0.04
Menthone Glycerol Ketal	0.05	0.10	0.20	0.35	0.05	0.04
WS-3		0.05	0.05			_
Menthyl Lactate	0.30	_	0.20	_		0.10
Menthyl Succinate		_	_	0.20	0.08	0.10
Menthol		0.15				0.25
	100.00	100.00	100.00	100.00	100.00	100.00

The Takasago cooling agent (TCA) may produce synergistic cooling effects when combined with menthol in chewing gum. It is also possible to take advantage of differing release rates of the two cooling agents to provide a fast cooling release and a long lasting cooling in the same product. Also, reductions in bitterness may be achieved through optimization of the levels of these cooling agents. For example, chewing gums may be prepared from the following formulas:

TABLE 28

	Example 106	Example 107
Gum Base	25.00%	30.00%
Sugar	56.76%	58.29%
Corn Syrup	15.00%	9.00%
Glycerin	2.00%	1.50%
Spearmint Oil	0.70%	
Peppermint Oil	<del></del>	0.90%
TCA	0.04%	0.06%
Menthol	0. <u>50%</u>	0.25%
TOTAL	100.00	100.00

10

Additional cooling agents may also be added as in some of the following examples:

TABLE 29

•	Example 108	Example 109	Example 110	Example 111	Example 112	Example 113
Gum Base	48.00	35.00	30.00	25.00	22.00	30.00
Sorbitol	33.85	48.60	53.12	60.69	59.54	50.46
Hydrogenated Starch Hydrolysate Syrup	_	-		7.50	14.00	10.00
70% Sorbitol Solution	10.00	8.00	_	<del></del>		
Glycerin	5.00	6.00	15.00	5.00	3.00	7.00
Encapsulated Aspartame	0.30	0.50	0.60	0.20	0.30	0.40
Methyl Salicylate	0.50	_	_		0.70	
Peppermint Oil	1.30	0.40	_	0.95	0.20	1.20
Spearmint Oil	0.20	0.80	0.80	_	_	
TCA	0.30	0.10	0.08	0.11	0.13	0.04
Menthol	0.25	0.40	0.15	0.35	0.05	0.45
WS-3		0.05	0.05			
Menthyl Lactate	0.30		0.20	_		0.10
Menthyl Succinate	·		_	0.20	0.08	0.10
Menthone Glycerol Ketal		0.15				0.25
•	100.00	100.00	100.00	100.00	100.00	100.00

The cooling agent 3-l-menthoxypropane-1,2-diol may produce synergistic cooling effects when combined with acyclic carboxamides, for example N-2,3-trimethyl-2-isopropyl butanamide (WS-23), in chewing gum. It is also possible to take advantage of differing release rates of the two cooling agents to provide a fast cooling release and a long lasting cooling in the same product. Also, reductions in bitterness may be achieved through optimization of the levels of these cooling agents. For example, chewing gums may be prepared from the following formulas:

TABLE 30

	Example 114	Example 115
Gum Base	25.00%	30.00%
Sugar	57.11%	58.46%
Com Syrup	15.00%	9.00%
Glycerin	2.00%	1.50%
Spearmint Oil	0.70%	_
Peppermint Oil	<del>-</del>	0.90%
WS-23	0.08%	0.06%
TCA	<u>0.11%</u>	_0.08%
TOTAL	100.00	100.00

10

Additional cooling agents may also be added as in some of the following examples:

TABLE 31

	Example 116	Example 117	Example 118	Example 119	Example 120	Example 121
Gum Base	48.00	35.00	30.00	25.00	22.00	30.00
Sorbitol	34.05	48.85	52.57	60.69	59.24	51.12
Hydrogenated Starch Hydrolysate Syrup	_	_	_	7.50	14.00	10.00
70% Sorbitol Solution	10.00	8.00		_		
Glycerin	5.00	6.00	15.00	5.00	3.00	7.00
Encapsulated Aspartame	0.30	0.50	0.60	0.20	0.30	0.40
Methyl Salicylate	0.50	_		_	0.70	_
Peppermint Oil	1.30	0.40	_	0.95	0.20	1.20
Spearmint Oil	0.20	0.80	0.80		_	
WS-3	_		0.08	0.16	_	
Menthone Glycerol Ketal			_	_	0.05	0.04
WS-23	0.30	0.05	0.05	0.20	0.13	0.04
Menthyl Lactate	0.30	0.10	0.20	0.10	_	0.10
TCA	0.05	0.10	0.20	0.20	0.08	0.10
Menthol		0.20	0.50		0.30	
	100.00	100.00	100.00	100.00	100.00	100.00

The cooling agent 3-I-menthoxypropane-1,2-diol may produce synergistic cooling effects when combined with N-substituted p-menthane carboxamides, for example N-ethyl-p-menthane-3-carboxamide (WS-3), in chewing gum. It is also possible to take advantage of differing release rates of the two cooling agents to provide a fast cooling release and a long lasting cooling in the same product. Also, reductions in bitterness may be achieved through optimization of the levels of these cooling agents. For example, chewing gums may be prepared from the following formulas:

TABLE 32

	Example 122	Example 123
Gum Base	25.00%	30.00%
Sugar	57.11%	58,46%
Corn Syrup	15.00%	9.00%
Glycerin	2.00%	1.50%
Spearmint Oil	0.70%	
Peppermint Oil	<u> </u>	0.90%
WS-3	0.08%	0.06%
TCA	_ 0.11%	0.08%
TOTAL	100.00	100.00

10

Additional cooling agents may also be added as in some of the following examples:

TABLE 33

	Example 124	Example 125	Example 126	Example 127	Example 128	Example 129
Gum Base	48.00	35.00	30.00	25.00	22.00	30.00
Sorbitol	34.05	48.85	52.57	60.69	59.24	51.12
Hydrogenated Starch	_	_	-	7.50	14.00	10.00
Hydrolysate Syrup						
70% Sorbitol Solution	10.00	8.00	_			_
Glycerin	5.00	6.00	15.00	5.00	3.00	7.00
Encapsulated Aspartame	0.30	0.50	0.60	0.20	0.30	0.40
Methyl Salicylate	0.50			_	0.70	_
Peppermint Oil	1.30	0.40		0.95	0.20	1.20
Spearmint Oil	0.20	0.80	0.80	_	_	
WS-3	0.30	0.10	0.08	0.25	0.13	0.04
Menthone Glycerol Ketal				0.05	0.05	0.04
WS-23	_	0.05	0.05	0.06	_	_
Menthyl Lactate	0.30		0.20	0.10		0.10
TCA	0.05	0.10	0.20	0.20	0.08	<b>0</b> .10
Menthol	_	0.20	0.50		0.30	
	100.00	100.00	100.00	100.00	100.00	100.00

The above tables show formulations in a peppermint flavored, spearmint flavored or wintergreen flavored chewing gum. Other gum products can be made with other flavors as well. Menthol and/or physiological cooling agents may enhance these various types of flavors, such as menthol-eucalyptus, spearmint-menthol, cinnamon-menthol, and even fruity mint-menthol.

The formulations for various flavored gum formulas are found in tables 34 and 35. Again, the cooling agents may (Examples 130-136) or may not be encapsulated, depending on whether a modified release is desired.

TABLE 34 **VARIOUS-FLAVORED SUGARLESS GUM** 

	Example 130	Example 131	Example 132	Example 133
Gum Base	30.00	30.00	30.00	30.00
Calcium Carbonate	5.00	5.00	5.00	5.00
Sorbitol	54.60	53.68	53.80	55.05
Glycerin	8.00	8.00	8.00	7.00
Encapsulated Sweetener	0.50	0.50	0.50	0.50
Flavor	1.00 <sup>A</sup>	1. <b>50<sup>B</sup></b>	1.40 <sup>B</sup>	1.60 <sup>C</sup>
Menthol	0.20	0.12	0.30	0.20
Active ML/PMCD	0.10	0.20	0.10	0.05
Lecithin	0.60	0.60	0.60	0.60
Encapsulated Menthol		0.40	0.30	_
TOTAL	100.00	100.00	100.00	100.00

TABLE 35 **VARIOUS-FLAVORED SUGARLESS GUM** 

	Example 134	Example 135	Example 136
Gum Base	30.00	30.00	30.00
Calcium Carbonate	5.00	5.00	5.00
Sorbitol	53.90	52.30	52.30
Glycerin	8.00	10.00	10.00
Encapsulated Sweetener	0.50	0.60	0.60
Flavor	1.60 <sup>C</sup>	1.20 <sup>D</sup>	1.20 <sup>D</sup>
Menthol	0.20	0.20	0.10
Active ML/Acyclic	0.20	0.10	0.20
Carboxamide (AC)			
Lecithin	0.60	0.60	0.60
Encapsulated Menthol			. —
TOTAL	100.00	100.00	100.00

<sup>\* 50/50</sup> blend of ML and acyclic carboxamide (AC) Cinnamon-Menthol Flavor Fruity-Mint Menthol Flavor

5 The combination of physiological cooling agents may be used in a wide variety of sugarless and sugar chewing gum formulations. The

<sup>&</sup>lt;sup>A</sup> Spearmint Flavor
<sup>B</sup> Menthol-Eucalyptus Flavor

<sup>&</sup>lt;sup>C</sup> Cinnamon-Menthol Flavor
<sup>D</sup> 50/50 blend of ML and PMC compounds

cooling agents may be encapsulated or entrapped in a wide variety of controlled release techniques as previously discussed. Gum formulations in which these materials may be used are given in tables 36-42. These formulas may also be made with non-encapsulated physiological cooling agents. Examples of the techniques and resulting controlled release physiological cooling agents that may be used in these formulations are discussed in the examples following the tables.

TABLE 36
Regular-Tack Sugarless Gum

			_		
	Example 137	Example 138	Example 139	Example 140	Example 141
Sorbitol	50.00	50.00	50.00	50.00	50.00
Gum Base	24.70	24.70	24.70	24.70	24.70
Lecithin	0.20	0.20	0.18	0.18	0.18
Glycerin	2.00	2.00	2.00	5.00	8.00
Lycasin	14.40	12.00	12.00	9.00	6.00
Mannitol	7.10	9.50	9.48	9.53	9.53
Peppermint Flavor	1.40	1.40	1.44	1.44	1.44
Active Level of Cooling Agents	0.20	0.20	0.20	0.15	0.15
TOTAL	100.00	100.00	100.00	100.00	100.00

TABLE 37
Regular-Tack Sugarless Gum

	Example 142	Example 143	Example 144	Example 145	Example 146
Sorbitol	49.35	49.35	49.35	49.35	49.35
Gum Base	25.50	25.50	25.50	25.50	25.50
Lecithin	0.20	0.20	0.20	0.20	0.20
Glycerin	8.50	8.50	8.50	8.60	8.50
Liquid Sorbitol	6.80	6.80	6.80	6.80	6.90
Mannitol	8.00	8.00	8.00	8.00	8.00
Active Level of Cooling Agents	0.20	0.20	0.20	0.10	0.10
Peppermint Flavor	1.45	1.45	1.45	1.45	1.45
TOTAL	100.00	100.00	100.00	100.00	100.00

TABLE 38
Sugarless Pellet Gums for Coating

	Example 147	Example 148	Example 149	Example 150	Example 151
Sorbitol	51.16	43.87	45.92	43.81	46.33
Gum Base	31.01	33.00	32.71	33.03	30.97
Glycerin	6.14	8.00	7.50	7.98	7.82
Aspartame	0.06		0.10	_	80.0
Active Level of Cooling Agents	0.42	0.23	0.35	0.25	0.34
Calcium Carbonate	10.01	13.00	12.16	12.93	13.04
Peppermint Flavor		1.20	0.17	*****	1.01
Menthol		0.50	_		0.21
Fruit Flavor		_		1.50	_
Lemon Flavor		_		.50	_
Encapsulated Menthol	_	0.20	_	<del></del>	0.20
Spearmint Flavor	1.20		1.09		_
TOTAL	100.00	100.00	100.00	100.00	100.00

TABLE 39
Sugarless Bubble Gums

	Example 152	Example 153	Example 154	Example 155
Sorbitol	56.65	56.09	50.42	48.63
Gum Base	24.00	24.59	28.00	30.10
Lecithin	1.00	0.91	0.89	0.86
Fruit Flavor	1.20	1.41	_	
Grape Flavor		_	1.71	
Strawberry Flavor	—	-	_	1.41
Evaporated Lycasin/Glycerin*		6.79	9.61	10.41
Glycerin	17.00	10.00	9.00	8.21
Free Aspartame	0.04		0.06	0.17
Active Level of Cooling Agents	0.11	0.21	0.31	0.21
TOTAL	100.00	100.00	100.00	100.00

<sup>\*</sup> Contains 25% glycerin, 67.5% Lycasin brand hydrogenated starch hydrolsate solids and 7.5% water.

TABLE 40 Sugar Gums

<u></u>	<del></del>				
	Example 156	Example 1 <b>57</b>	Example 158	Example 159	Example 160
Sugar	58.29	59.26	62.49	59.97	56.61
Gum Base	22.38	20.60	20.08	23.17	26.80
Com Syrup	17.20	18.50	15.40	14.70	13.88
Glycerin	1.09	0.83	1.00	1.00	1.30
Active Level of Cooling Agents	0.10	0.20	0.15	0.25	0.20
Lecithin	0.05	0.03	0.02		
Peppermint Flavor	0.89	0.58	0.86	0.91	1.21
TOTAL	100.00	100.00	100.00	100.00	100.00

TABLE 41 Sugar Gums

	Example 161	Example 162	Example 163	Example 164	Example 165
Sugar	54.30	45.30	49.30	40.30	45.30
Gum Base	19.20	19.20	19.20	19.20	19.20
Glycerin	1.40	1.40	1.40	1.40	1.40
Corn Syrup	19.00	23.00	19.00	19.00	23.00
Dextrose	_	5.00	_	_	_
Lactose	5.00	· ·	_		
Fructose		5.00		_	
Invert Sugar		_	10.00	_	_
Maltose	_		_	10.00	
Palatinose	_		_		10.00
Spearmint Flavor	0.90	0.90	0.90	9.90	0.90
Active Level of Cooling Agents	0.20	0.20	0.20	0.20	0.20
TOTAL	100.00	100.00	100.00	100.00	100.00

TABLE 42 Sugarless Gums

	Example 166	Example 167	Example 168	Example 169	Example 170	Example 171	Example 172	Example 173
Gum Base	25.50	25.50	25.50	25.50	25.50	25.50	25.50	25.50
Sorbitol	53.80	46,80	41.80	41.80	41.80	41.80	36.80	37.80
Sorbitol Liquid/ Lycasin	17.00	14.00	6.00	_	5.00		_	11.00 <sup>A</sup>
Mannitol	_	10.00	8.00	8.00	8.00	8.00	8.00	8.00
Maltitol	_	_	_	5.00	_		5.00	_
Xylitol	_		15.00	10.00	_	_	5.00	10.00
Lactitol	_	_	_	_	10.00		_	
Hydrogenated Isomaltulose	-	_	_		_	15.00	10.00	-
Giycerin	2.00	2.00	2.00	8.00	8.00	8.00	8.00	6.00
Flavor	1.50	1.50	1.50	1.50	1.50	1.50	1.50	1.50
Active Level of Cooling Agents	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20
TOTAL	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00

<sup>&</sup>lt;sup>A</sup> Lycasin brand hydrogenated starch hydrolsate syrup; all others use 70% sorbitol liquid.

Encapsulated cooling agents may be made by the Examples 174-226 that follow and added to any of the formulas given in tables 36 through 42. Encapsulations with water soluble polymers such as HPMC or maltodextrins will give a fast release of cooling agent. Encapsulations with shellac, Zein or PVAC will give a slow release.

## Example 174

5

10

This example contains a cooling agent composition which has 25% menthyl succinate and 75% PMC entrapped with polyvinyl-acetate.

## Example 175

This example contains a cooling agent composition containing 50% menthyl succinate and 50% PMC agglomerated with hydroxypropylmethyl cellulose.

This example contains a cooling agent composition which has 75% menthyl succinate and 25% PMC coated with Zein.

## Example 177

This example contains a cooling agent composition which has 25% menthyl lactate and 75% MGK absorbed onto silica.

## Example 178

This example contains a cooling agent composition which has 50% menthyl succinate and 50% MGK coated with shellac.

## 10 **Example 179**

This example contains a cooling agent composition which has 25% menthyl succinate and 75% 3-l-menthoxypropane-1,2-diol (TCA) extruded with polyvinyl acetate.

## Example 180

This example contains a cooling agent composition which has 50% menthyl succinate and 50% 3-l-menthoxypropane-1,2-diol (TCA) coated with shellac.

#### Example 181

20

This example contains a cooling agent composition which has 75% menthyl succinate and 25% 3-l-menthoxypropane-1,2-diol (TCA) coated with Zein.

This example contains a cooling agent composition which has 25% menthyl succinate and 75% MGK extruded with polyvinyl acetate.

#### Example 183

This example contains a cooling agent composition which has 50% menthyl succinate and 50% MGK coated with Zein.

### Example 184

This example contains a cooling agent composition which has 75% menthyl succinate and 25% MGK agglomerated with hydroxypropylmethyl cellulose.

### Example 185

10

This example contains a cooling agent composition which has 25% menthyl succinate and 75% acyclic carboxamide (AC) coated with Zein.

## Example 186

This example contains a cooling agent composition which has 50% menthyl succinate and 50% acyclic carboxamide (AC) coated with hydroxypropylmethyl cellulose.

#### Example 187

This example contains a cooling agent composition which has 75% menthyl succinate and 25% acyclic carboxamide (AC) absorbed onto silica.

# Example 188

This example contains a cooling agent composition which has 25% ML and 75% 3-l-menthoxypropane-1,2-diol (TCA) agglomerated with gelatin.

This example contains a cooling agent composition which has 50% ML and 50% 3-l-menthoxypropane-1,2-diol (TCA) absorbed onto silica.

## Example 190

This example contains a cooling agent composition which has 75% ML and 25% 3-l-menthoxypropane-1,2-diol (TCA) coated with shellac.

### Example 191

This example contains a cooling agent composition which has 50% ML and 50% acyclic carboxamide (AC) which is agglomerated with Zein.

#### 10 **Example 192**

This example contains a cooling agent composition which has 25% ML and 75% acyclic carboxamide (AC) which is agglomerated with hydroxypropylmethyl cellulose.

## Example 193

This example contains a cooling agent composition which has 75% ML and 25% acyclic carboxamide (AC) coated with shellac.

#### Example 194

20

This example contains a cooling agent composition which has 50% menthyl lactate and 50% p-menthane carboxamide (PMC) coated with maltodextrin.

This example contains a cooling agent composition which has 25% 3-l-menthoxypropane-1,2-diol and 75% acyclic carboxamide (AC) extruded with polyvinyl acetate.

## 5 Example 196

This example contains a cooling agent composition which has 50% 3-l-menthoxypropane-1,2-diol and 50% p-menthane carboxamide (PMC) agglomerated with Zein.

## Example 197

An 80% shellac, 20% active cooling agent powder mixture is obtained by spray drying an alcohol/shellac/menthyl succinate and menthyl lactate solution.

#### Example 198

A 50% shellac, 50% active cooling agent powder mixture is obtained

by spray drying an appropriate ratio of alcohol/shellac/menthyl succinate and
menthyl lactate solution.

### Example 199

A 70% Zein, 30% active cooling agent powder mixture is obtained by spray drying an alcohol/shellac/menthyl succinate and menthyl lactate solution.

### Example 200

20

A shellac/silica/active cooling agent powder mixture is obtained by fluid-bed coating menthyl lactate and MGK absorbed on silica with an alcohol/shellac solution at 20% solids.

A shellac/silica/active cooling agent powder mixture is obtained by fluid-bed coating menthyl succinate and 3-l-menthoxypropane-1,2-diol (TCA) absorbed on silica with an alcohol/shellac solution at 20% solids.

#### 5 **Example 202**

A Zein/silica/active cooling agent mixture is obtained by fluid-bed coating menthyl succinate and menthyl lactate absorbed on silica with an alcohol/Zein solution at 25% solids.

#### Example 203

An 85% wax, 15% active PMC and AC powder mixture is obtained by spray chilling a mixture of molten wax and cooling agent.

### Example 204

A 70% wax, 30% active PMC and AC powder mixture is obtained by spray chilling a mixture of molten wax and cooling agent.

## 15 **Example 205**

A 70% Zein, 30% active menthyl succinate and p-menthane carboxamide (PMC) powder mixture is obtained by spray drying an aqueous mixture of cooling agent and Zein dispersed in an aqueous, high-pH (pH = 11.6-12.0) media at 15% solids.

#### 20 **Example 206**

A Zein/active menthyl succinate and p-menthane carboxamide (PMC) powder mixture is obtained by fluid-bed coating cooling agent with an aqueous, high-pH (pH = 11.6-12.0) Zein dispersion of 15% solids.

10

### Example 207

A 20% Zein, 20% shellac, 60% active menthyl succinate and AC powder mixture is obtained by spray drying an alcohol/shellac/cooling agent mixture and then fluid-bed coating the spray dried product for a second coating of alcohol and Zein.

Examples 197-207 would all give nearly complete encapsulation and would delay the release of the cooling agents when used in gum formulations in tables 36 through 42. The higher levels of coating would give a longer delayed release of the cooling agents than the lower levels of coating.

Other polymers that are more water soluble and used in coating would have less of an effect of delaying the release of the cooling agents.

## Example 208

An 80% gelatin, 20% active PMC and TCA compounds powder mixture is obtained by spray drying a gelatin/TCA and PMC compounds emulsion.

## 15 **Example 209**

A 50% hydroxypropylmethyl cellulose (HPMC), 50% active menthyl succinate and PMC compounds powder mixture is obtained by fluid-bed coating menthyl succinate and PMC compounds with an aqueous solution of HPMC at 10% solids.

## 20 Example 210

A 30% ethyl cellulose, 70% active menthyl succinate and PMC compounds powder mixture is obtained by fluid-bed coating menthyl succinate and PMC compounds with an aqueous solution of ethyl cellulose at 10% solids.

A 50% maltodextrin, 50% active TCA and AC compounds powder mixture is obtained by spray drying an aqueous emulsion of TCA and AC compounds and maltodextrin at 40% solids.

## 5 **Example 212**

10

15

A 50% gum arabic, 50% active TCA and AC compounds powder mixture is obtained by fluid-bed coating TCA and AC compounds absorbed on silica, then with an aqueous solution of gum arabic at 40% solids.

The coated TCA and PMC compounds from Example 208, and the coated menthyl succinate and PMC compounds of Examples 209 and 210, when used in the chewing gum formula in tables 36 through 42, would give a moderately fast release of cooling agents. The products coated with maltodextrin and gum arabic in Examples 211 and 212, when used in the gum formula in tables 36 through 42, would give a fast release of the cooling agents.

Cooling agents could also be used in gum after being agglomerated to give modified release of these cooling agents.

## Example 213

A 15% hydroxypropylmethyl cellulose (HPMC), 85% active TCA and
PMC compounds powder mixture can be prepared by agglomerating TCA and
PMC compounds and HPMC blended together, with water being added, and
the resulting product being dried and ground.

# Example 214

A 15% gelatin, 85% active TCA and PMC compounds powder mixture

can be made by agglomerating TCA and PMC compounds and gelatin

blended together, with water being added, and the resulting product being dried and ground.

A 10% Zein, 90% active TCA and AC compounds powder mixture can be made by agglomerating TCA and AC compounds with an aqueous solution containing Zein, and drying and grinding the resulting product.

## 5 **Example 216**

A 15% shellac, 85% active TCA and AC compounds powder mixture can be made by agglomerating TCA and AC compounds with an alcohol solution containing 25% shellac, and drying and grinding the resulting product.

10

15

20

Examples of multiple step treatments are here described:

## Example 217

Menthyl succinate and TCA are spray dried with maltodextrin at 30% solids to prepare a powder. This powder is then agglomerated with a hydroxypropylmethyl cellulose (HPMC) in a ratio of 85/15 powder/HPMC, wetted with water and dried. After grinding the resulting powder will contain about 68% active cooling agent, 17% maltodextrin and 15% HPMC.

#### Example 218

Menthyl succinate and ML is agglomerated with HPMC in a ratio of 85/15 cooling agent/HPMC. After drying and grinding, the resulting powder is fluid-bed coated with an alcohol shellac solution at about 25% solids to give a final product containing about 60% active cooling agent, 10% HPMC, and about 30% shellac.

#### Example 219

Menthyl succinate and ML is agglomerated with HPMC in a ratio of 85/15 cooling agent/HPMC. After drying and grinding, the resulting powder is

agglomerated with a 15% solids, high-pH, aqueous solution of Zein to give a final product containing about 60% active cooling agent, 10% HPMC, and 30% Zein.

## Example 220

Menthyl lactate and TCA is spray dried with a 25% emulsion of gelatin. The spray dried product is then agglomerated with a 15% solids, high-pH, aqueous solution of Zein. The final product will contain about 50% active cooling agent, 20% gelatin, and 30% Zein.

#### Example 221

Menthyl succinate and AC is agglomerated with molten wax in a ratio of 85/15 cooling agent/wax. When the mixture cools and is ground, it is fluid-bed coated with a 10% Zein solution, giving a final product containing 60% active cooling agent, 10% wax, and 30% Zein.

#### Example 222

A mixture of MGK and TCA is sprayed onto a precipitated silica. The mixture is dried and ground. The final product is about 50% active cooling agent.

#### Example 223

20

25

A mixture of MGK and TCA is sprayed onto a pharmasorb clay. The mixture is dried and ground and gives a final product of about 80% clay and 20% active cooling agent.

#### Example 224

A mixture of MGK and TCA is sprayed onto a microcrystalline cellulose powder. The mixture is dried and ground and gives a product that is about 70% microcrystalline cellulose and 30% active cooling agent.

A mixture of MGK and TCA is sprayed onto a high absorption starch. The mixture is dried and ground and gives a product that is about 80% starch and 20% active cooling agent.

# 5 **Example 226**

10

15

20

A mixture of MGK and TCA is sprayed onto a calcium carbonate powder. The mixture is dried and ground and gives a product of about 90% calcium carbonate and 10% active cooling agent.

Many of the examples listed are single step processes. However, more delayed release of the cooling agents may be obtained by combining the various processes of encapsulation, agglomeration, absorption, and entrapment. Any of the above preparations can be further treated in fluid-bed coating, spray chilling or coacervation processes to encapsulate the product, and can be agglomerated with various materials and procedures in a variety of multiple step processes.

As disclosed in many of the patents, physiological cooling agents may be added to chewing gum formulations. These chewing gums may be used as centers or cores for the chewing gum that are coated. TABLE 43 illustrates sugared chewing gum formulations that are used as centers for sugar-coated chewing gums having a coating comprising a physiological cooling agent.

TABLE 43
SUGARED CHEWING GUM CENTERS

SUGARED CHEWING GUM CENTERS				
	Example 227	Example 228	Example 229	
Gum Base	28.0%	30.0%	30.0%	
Sugar	52.9%	50.7%	46.6%	
Corn Syrup	18.0%	18.0%	12.0%	
Glycerin	0.5%	0.5%	0.5%	
Spearmint Flavor	0.6%	_		
Peppermint Flavor		0.8%	0.9%	
Dextrose Monohydrate		_	10.0%	

The chewing gum formed in TABLE 43 is sheeted in pellets that are square or rectangular pillow shaped and coated with the sugar-based coating formulations in which the cooling agent and menthol are dissolved in the flavor when mixed into the coating syrup in TABLE 44.

TABLE 44
COATING FOR SUGARED CHEWING GUM CENTERS

5

10

15

	Example 230	Example 231	Example 232
Sugar	96.56	96.06	95.71
Modified Starch	3.0	3.0	3.0
Spearmint Flavor	0.3	-	-
Peppermint Flavor		0.6	0.8
Menthol	•	0.2	0.3
Camauba Wax	0.04	0.04	0.04
Cooling Agent	0.1	0.1	0.15

<sup>\*</sup>Any of the cooling agents listed such as PMC, AC, ML, MS, MGK, TCA or combinations may be used.

In Example 230, the physiological cooling agent gives a clean, cool Spearmint flavor that could not be obtained with menthol. Examples 231 and 232 would normally require higher levels of menthol, and consequently would contain harsh notes. The addition of the physiological cooling agents to Examples 231 and 232 result in strong, clean, cool minty tasting products.

As stated previously, non-sugar or sugarless coatings generally will have less sweetness, thus causing more harsh notes when mint flavor and/or menthol are added to the coating. Use of the physiological cooling agents is especially useful in sugarless coating. Polyols such as sorbitol, xylitol, maltitol, lactitol and hydrogenated isomaltulose can be used to coat pellet sugarless gum. Examples of sugarless gum center or core formulations are found in tables 45 and 46.

TABLE 45
SUGARLESS GUM CENTERS

	Example 233	Example 234	Example 235	Example 236	
Base	33.1	30.0	30.0	33.0	
Calcium Carbonate	15.0	11.0	11.0	13.0	
Sorbitol	40.94	46.36	46.08	49.52	
Glycerin	8.0	0.4	0.4	0.4	
Liquid Sorbitol	_	6.2	6.2	_	
Encap. Sweetener	0.7	0.24	0.24	0.82	
Wintergreen Flavor	1.1				
Menthol	1.1		0.26	0.3	
Cooling Agent*	0.06		0.12	0.06	
Mannitol	_	3.0	3.0	-	
Peppermint Flavor	_	2.0	1.9	1.9	
Lecithin	_	0.8	8.0	1.0	
Encap Menthol	_			_	

<sup>\*</sup> Any of the cooling agents listed such as PMC, AC, ML, MS, MGK, TCA or combinations may be used.

TABLE 46 SUGARLESS GUM CENTERS

8	_	

	Example 237	Example 238	Example 239
Base	32.0	31.6	33.6
Calcium Carbonate	15.0	11.0	13.0
Sorbitol	41.26	45.98	49.23
Glycerin	7.0	8.0	0.75
Liquid Sorbitol	_	7.2	
Encap. Sweetener	0.40	0.66	0.66
Wintergreen Flavor	_	-	_
Menthol	0.9	0.3	0.3
Cooling Agents*	0.14	0.06	0.08
Mannitol	_		_
Peppermint Flavor	1.3	1.9	1.9
Lecithin	_	0.5	0.5
Encap Menthol	2.0	_	_

 $<sup>^{\</sup>star}$  Any of the cooling agents listed such as PMC, AC, ML, MS, MGK, TCA or combinations may be used.

The centers in tables 45 and 46 are coated with coating formulations that comprise xylitol, maltitol, lactitol, hydrogenated isomaltulose or sorbitol; various flavors; optionally menthol; and the physiological cooling agents. Examples of sugarless coated chewing gums are found in tables 47 and 48.

5

10

TABLE 47
SUGARLESS COATED CHEWING GUMS

	Ex. 240	Ex. 241	Ex. 242	Ex. 243	Ex. 244
Center	Ex. 233	Ex. 234	Ex. 234	Ex. 235	Ex. 236
Xylitol	88.83	88.83		_	_
Gum Arabic	8.8	8.8	2.8	2.8	3.1
Titanium Dioxide	0.9	0.9	0.9	0.9	1.3
Hydrogenated isomaltulose			95.0	94.84	93.66
Maltitol		_	_		
Wintergreen Flavor	0.9		_		
Peppermint Flavor		0.9	0.7	0.7	0.75
Menthol	0.4	0.4	0.3	0.5	0.75
Carnauba Wax	0.1	0.1	0.1	0.1	0.1
Cooling Agent*	0.07	0.07	0.1	0.06	0.14
Sweetener	_		0.1	0.1	0.2

<sup>\*</sup>Cooling agent used in the examples may contain ML, MS, MGK, AC, PMC, TCA or combinations

TABLE 48
SUGARLESS COATED CHEWING GUMS

SUGARLESS COATED CHEVAING GOWS						
	Ex. 245	Ex. 246	Ex. 247	Ex. 248		
Center	Ex. 236	Ex. 237	Ex. 238	Ex. 239		
Xylitol	_		_			
Gum Arabic	3.1	3.1	3.1	3.1		
Titanium Dioxide	1.3	1.3	1.3	1.3		
Hydrogenated Isomaltulose			93.59	93.56		
Maltitol	93.66	93.33	_	****		
Wintergreen Flavor		_	_			
Peppermint Flavor	0.75	1.0	0.9	0.9		
Menthol	0.75	1.0	0.3	0.3		
Carnauba Wax	0.1	0.1	0.1	0.1		
Cooling Agent*	0.14	0.02	0.11	0.14		
Sweetener	0.2	0.15	0.6	0.6		

<sup>\*</sup>Cooling agent used in the examples may contain ML, MS, MGK, AC, PMC, TCA or combinations

In the examples of tables 47 and 48, menthol is dissolved into the flavor along with the physiological cooling agent. Half of this mixture is

10

applied at each of coats 8 and 14. After the coating is completed and allowed to stand overnight, the pellets are polished with carnauba wax. In the case of hydrogenated isomaltulose and maltitol, gum arabic may be used as a precoat with hydrogenated isomaltulose or maltitol, or dusted with powdered hydrogenated isomaltulose or maltitol then coated with a hydrogenated isomaltulose or maltitol solution.

Other coated gum products can be made with other flavors as well. Menthol and/or physiological cooling agents may enhance these various types of flavors such as menthol-eucalyptus, spearmint-menthol, cinnamon-menthol, and even fruity mint-menthol. The formulations for various flavored centers are found in tables 49 and 50.

TABLE 49
VARIOUS-FLAVORED CENTERS

	Ex. 249	Ex. 250	Ex. 251	Ex. 252
Base	30.0	30.0	30.0	30.0
Calcium Carbonate	11.0	11.0	11.0	11.0
Sorbitol	51.6	50.6	50.8	52.05
Glycerin	5.0	5.0	5.0	4.0
Encap. Sweetener	0.5	0.5	0.5	0.5
Flavor	1.0 <sup>A</sup>	1.5 <sup>B</sup>	1.4 <sup>B</sup>	1.6 <sup>C</sup>
Menthol	0.2	0.4	0.3	0.2
Cooling agent*	0.1		0.1	0.05
Lecithin	0.6	0.6	0.6	0.6
Encap. Menthol	·	0.4	0.3	

<sup>&</sup>lt;sup>A</sup>Spearmint Flavor

<sup>&</sup>lt;sup>B</sup>Menthol-Eucalyptus Flavor

<sup>&</sup>lt;sup>C</sup>Cinnamon-Menthol Flavor

<sup>\*</sup>Cooling agent used in the examples may contain ML, MS, MGK, AC, PMC,

TCA or combinations

TABLE 50 **VARIOUS-FLAVORED CENTERS** 

	Ex. 253	Ex. 254	Ex. 255
Base	30.0	30.0	30.0
Calcium Carbonate	11.0	11.0	11.0
Sorbitol	51.9	49.3	49.3
Glycerin	4.0	7.0	7.0
Encap. Sweetener	0.5	0.6	0.6
Flavor	1.6 <sup>C</sup>	1.2 <sup>D</sup>	1.2 <sup>D</sup>
Menthol	0.2	0.2	0.1
Cooling Agent*	0.2	0.1	0.2
Lecithin	0.6	0.6	0.6
Encap. Menthol		_	_

<sup>&</sup>lt;sup>C</sup>Cinnamon-Menthol Flavor <sup>D</sup>Fruity-mint Menthol Flavor

TCA or combinations

5

The various-flavored center formulas may be coated with polyols such as xylitol, maltitol, lactitol, hydrogenated isomaltulose or sorbitol and have a physiological cooling agent added to the coating. Examples of coated chewing gum having various flavors are found in tables 51 and 52.

TABLE 51 **VARIOUS-FLAVORED COATED CHEWING GUMS** 

	Ex. 256	Ex. 257	Ex. 258	Ex. 259
Center	Ex. 249	Ex. 250	Ex. 251	Ex. 252
Xylitol	89.7	89.1	_	89.2
Gum Arabic	8.8	8.8	2.8	8.8
Titanium Dioxide	0.8	8.0	0.8	0.8
Hydrogenated isomaltulose			95.0	_
Maltitol	_	_		-
Flavor	0.4 <sup>A</sup>	0.6 <sup>8</sup>	0.6 <sup>8</sup>	0.8 <sup>C</sup>
Menthol	0.1	0.4	0.3	0.2
Camauba Wax	0.1	0.1	0.1	0.1
Cooling Agent	0.1	0.2	0.2	0.1
Sweeteners		_	0.2	

<sup>\*</sup>Cooling agent used in the examples may contain ML, MS, MGK, AC, PMC,

ASpearmint Flavor

BMenthol-Eucalyptus Flavor

<sup>&</sup>lt;sup>C</sup>Cinnamon-Menthol Flavor

<sup>\*</sup>Cooling agent used in the examples may contain ML, MS, MGK, AC, PMC, TCA or combinations

TABLE 52
VARIOUS-FLAVORED COATED CHEWING GUMS

•	Ex. 260	Ex. 261	Ex. 262
Center	Ex. 253	Ex. 254	Ex. 255
Xylitol	_	89.7	
Gum Arabic	2.8	8.8	2.8
Titanium Dioxide	8.0	0.8	0.8
Hydrogenated isomaltulose	94.9	_	
Maltitol	_		95.6
Flavor	0.8 <sup>C</sup>	0.3 <sup>D</sup>	0.3 <sup>D</sup>
Menthol	0.2	0.1	0.1
Camauba Wax	0.1	0.1	0.1
Cooling Agent	0.2	0.2	0.1
Sweeteners	0.2	_	0.2

Cinnamon-Menthol Flavor

## **Breath Freshening Examples**

To evaluate chewing gum formulations made with a cinnamon flavor and low levels of mint flavor, along with physiological cooling agents for breath freshening, the following samples were made:

**TABLE 53** 

	Example 263 (comparative)	Example 264	Example 265	Example 266
Gum Base	19.46%	19.46%	19.46%	19.46%
Cinnamon Flavor	1.29%	1.29%	1.29%	1.39%
Peppermint Flavor	0.10%	0.10%	0.10%	
WS-3	-	0.10%	0.10%	0.10%
Menthone Glycerol Ketal	-		0.20%	<del>-</del>
Sugar	62.24%	62.14%	61.94%	62.14%
Corn Syrup	15.57%	15.57%	15.57%	15.57%
Glycerin	0.67%	0.67%	0.67%	0.67%
Color	0.67%	0.67%	0.67%	_0.67%
TOTAL	100.00	100.00	100.00	100.00

Informal evaluation showed Examples 263, 264 and 265 samples to have a clean cinnamon flavor with only a slight mint note. However,

10 Examples 264, 265 and 266 had noticeably higher cooling.

Pruity-mint Menthol Flavor

<sup>\*</sup>Cooling agent used in the examples may contain ML, MS,

MGK, AC, PMC, TCA or combinations

10

15

A trained panel of eight chewers were given a cracker smeared with garlic/onion cream cheese. They then chewed the gums of Examples 263 (comparative) and 264 (inventive) and rated "Fresh Breath", "Odor" and "Effectiveness for Breath Freshening" at 0.5, 1, 2, 3, 4, 5, 6, 7, 9, 11, 13 and 15 minutes. The gum was discarded and additional ratings were made at 5 and 10 minutes afterward (P5, P10). As can be seen from the graphs of FIGS. 1-3, which show the test results, the inventive composition had significantly better scores for each of the three rated properties.

In FIG. 1 it can be seen that the inventive gum (Example 264) produced generally higher scores for breath freshness. FIG. 2 rates the odor of the breath, and the Example 264 inventive gum produced lower odor ratings than the comparative gum (Example 263). FIG. 3 shows that the effectiveness for breath freshening was considered generally higher for the inventive gum (Example 264) than for the comparative gum (Example 263).

The following formulas are also illustrative of the invention.

TABLE 54

	IABLE :	04	
	Example 267	Example 268	Example 269
Gum Base	24.00%	24.00%	24.00%
Sorbitol	55.10%	55.00%	55.00%
Mannitol	7.00%	7.00%	7.00%
Hydrogenated Starch	6.50%	6.50%	6.50%
Hydrolysate Syrup			•
Glycerin	5.00%	5.00%	5.00%
Cinnamon Flavor	1.30%	1.30%	1.30%
WS-3	. <del></del>	0.10%	0.20%
Menthone Glycerol Ketal	0.20%	0.20%	0.10%
Red Color	0.50%	0.50%	0.50%
Aspartame	0.15%	0.15%	0.15%
Encapsulated Aspartame	0.25%	0.25%	0.25%
TOTAL	100.00	100.00	100.00

10

15

20

The samples will have a clean cinnamon flavor and enhanced breath freshening.

TABLE 55				
	Example 270	Example 271	Example 272	
Gum Base	24.00%	24.00%	24.00%	
Sorbitol	55.00%	55.00%	54.90%	
Mannitol	7.00%	7.00%	7.00%	
Hydrogenated Starch	6.50%	6.50%	6.50%	
Hydrolysate Syrup				
Glycerin	5.00%	5.00%	5.00%	
Cinnamon Flavor	1.30%	1.30%	1.30%	
WS-23		0.10%	0.20%	
Menthyl Lactate	0.20%	0.20%	0.10%	
Red Color	0.50%	0.50%	0.50%	
Aspartame	0.15%	0.15%	0.15%	
Encapsulated Aspartame	0.25%	0.25%	0.25%	
Menthyl Succinate	0.10%	_	0.10%	
TOTAL	100.00	100.00	100.00	

The samples will have a clean cinnamon flavor and enhanced breath freshening.

It should be appreciated that the methods and compositions of the present invention are capable of being incorporated in the form of a variety of embodiments, only a few of which have been illustrated and described above. The invention may be embodied in other forms without departing from its spirit or essential characteristics. It will be appreciated that the addition of some other ingredients, process steps, materials or components not specifically included will have an adverse impact on the present invention. The best mode of the invention may therefore exclude ingredients, process steps, materials or components other than those listed above for inclusion or use in the invention. However, the described embodiments are to be considered in all respects only as illustrative and not restrictive, and the scope of the invention is, therefore, indicated by the appended claims rather than by the foregoing description. All changes which come within the meaning and range of equivalency of the claims are to be embraced within their scope.

## **Claims**

- A method of producing chewing gum with physiological cooling agents comprising the steps of:
- a) mixing at least two physiological cooling agents together with a release modification material;
  - b) treating the cooling agents to provide a modified release; and
  - c) adding a quantity of the modified release cooling agents to a chewing gum composition to provide a cooling agent level in the gum of from about 0.001% to about 2%.

10

5

2. The method of claim 1 wherein the physiological cooling agents are selected from the group consisting of N-substituted p-menthane carboxamide, acyclic carboxamide, menthyl lactate, menthone glycerol ketal, 3-l-menthoxypropane-1,2-diol, menthyl succinate and combinations thereof.

- 3. The method of claim 1 wherein the cooling agents are treated by an encapsulation process.
- 4. The method of claim 3 wherein the cooling agents are encapsulated by a spray drying process.
  - 5. The method of claim 3 wherein the cooling agents are encapsulated by a fluid bed coating process.
- 25 6. The method of claim 1 wherein the cooling agents are treated by being entrapped in an extrusion process.
  - 7. A chewing gum made according to the method of claim 1.

WO 99/13734 PCT/US97/16731

78

8. A cooling flavor composition which imparts a physiological cooling effect to oral compositions in which it is used, but does not impart a peppermint flavor thereto, comprising:

about 40% to about 80% by weight menthol;

about 15% to about 50% by weight menthone; and
about 3% to about 25% by weight of at least one physiological cooling
agent selected from the group consisting of menthyl succinate; menthyl
lactate; 3-l-menthoxypropane-1,2-diol; N-substituted p-menthane
carboxamide; acyclic carboxamide; menthone glycerol ketals and mixtures
thereof.

- 9. The cooling flavor composition of claim 8 wherein the composition comprises at least two physiological cooling agents selected from the group consisting of menthyl succinate; menthyl lactate; 3-l-menthoxypropane-1,2-diol; N-substituted p-menthane carboxamide; acyclic carboxamide; menthone glycerol ketals and mixtures thereof.
- The cooling flavor composition of claim 9 wherein said at least two physiological cooling agents comprise, by weight of the cooling flavor
   composition:
  - a) about 1% to about 10% menthyl lactate, and
  - b) about 2% to about 15% menthyl succinate.
- 11. A chewing gum comprising a flavor and a cooling flavorcomposition according to claim 8.
  - 12. A coated chewing gum, the coating comprising:
    - a) a coating material and
    - b) a physiological cooling agent.

10

15

- 13. The coated chewing gum of claim 12 wherein the physiological cooling agent is selected from the group consisting of substituted pmenthanes, substituted pmenthane carboxamides, substituted cyclohexanamides, substituted cyclohexane carboxamides, substituted ureas, substituted sulphonamides, substituted menthanols, menthyl succinates and combinations thereof.
- 14. The coated chewing gum of claim 12 wherein the physiological cooling agent is selected from the group consisting of N-ethyl-p-menthane-3-carboxamide, acyclic carboxamide, menthyl lactate, menthyl succinate, 3-l-menthoxypropane-1,2-diol and combinations thereof.
  - 15. The coated chewing gum of claim 12 wherein the coating comprises about 0.001% to about 1.0% physiological cooling agent.

16. The coated chewing gum of claim 12 wherein the coating comprises about 0.01% to about 0.5% physiological cooling agent.

- 17. The coated chewing gum of claim 12 wherein the coating comprises about 0.02% to about 0.2% physiological cooling agent.
  - 18. The coated chewing gum of claim 12 wherein the coating further comprises a flavor.
- 25 19. The coated chewing gum of claim 18 wherein the flavor is selected from the group consisting of spearmint, peppermint, cinnamon, eucalyptus, fruity-mint, menthol, wintergreen and combinations thereof.
- 20. The coated chewing gum of claim 12 wherein the coating is a 30 soft coating.

10

- 21. A coated chewing gum, the coating comprising:
  - a) a coating material;
  - b) a physiological cooling agent; and
- c) a cooling flavor.
  - 22. The coated chewing gum of claim 21 wherein the coating comprises about 0.001% to about 1.0% physiological cooling agent and about 0.1% to about 1.0% cooling flavor.
- 23. The coated chewing gum of claim 21 wherein the coating comprises about 0.02% to about 0.2% physiological cooling agent.
- The coated chewing gum of claim 21 wherein the cooling flavor
   is selected from the group consisting of peppermint, eucalyptus, fruity-mint, menthol, wintergreen and combinations thereof.
  - 25. A coated chewing gum, the coating comprising
    - a) a coating material comprising a polyol; and
- 20 b) a physiological cooling agent.
  - 26. The coated chewing gum of claim 25 wherein the polyol is selected from the group consisting of xylitol, maltitol, lactitol, erythritol, sorbitol, hydrogenated isomaltulose and combinations thereof.
  - 27. The coated chewing gum of claim 25 wherein the polyol is selected from the group consisting of maltitol, lactitol, erythritol, sorbitol, hydrogenated isomaltulose and combinations thereof.

PCT/US97/16731

- 28. A method of improving the flavor impact of a coated chewing gum comprising the step of adding a physiological cooling agent to the coating.
- 5 29. The method of claim 28 further comprising the step of adding a cooling flavor to the coating.
  - 30. The method of claim 28 wherein the physiological cooling agent is selected from the group consisting of substituted p-menthanes, substituted p-menthane carboxamides, substituted cyclohexanamides, acyclic carboxamides, substituted cyclohexane carboxamides, substituted ureas, substituted sulphonamides, substituted menthanols, menthyl succinates and combinations thereof.
- 15 31. The method of claim 28 further comprising the step of adding to the coating a material selected from the group consisting of maltitol, lactitol, erythritol, sorbitol, hydrogenated isomaltulose and combinations thereof.
- 32. The method of claim 29 wherein the cooling flavor is selected from the group consisting of peppermint, eucalyptus, fruity-mint, menthol, wintergreen and combinations thereof.
- 33. The method of claim 28 wherein the physiological cooling agent is selected from the group consisting of N-ethyl-p-menthane-3-carboxamide,
   25 acyclic carboxamide, menthyl lactate, menthyl succinate, 3-l-menthoxypropane-1,2-diol and combinations thereof.

- 34. A method of coating a chewing gum comprising the steps of:
  - a) providing a chewing gum center; and
  - b) coating the center with a coating comprising:
    - i) a coating material; and

- ii) a physiological cooling agent.
- 35. The method of claim 34 wherein the coating material is a polyol other than xylitol.
- 10 36. A coated chewing gum having a clean cooling sensory perception comprising:
  - a) a chewing gum center; and
  - b) a coating comprising:
    - i) a coating material; and

- ii) a physiological cooling agent.
- 37. The coated chewing gum of claim 36 wherein the coating material is a polyol other than xylitol.
- 20 38. A method of replacing xylitol in a sugarless coated chewing gum comprising the steps of:
  - a) replacing at least some of the xylitol in the coating with a polyol that is less expensive than xylitol; and
    - b) adding a physiological cooling agent to the coating.

- 39. A high flavor-impact coated chewing gum comprising:
  - a) a chewing gum center; and
  - b) a coating comprising:
    - a coating material;
    - ii) a physiological cooling agent; and
    - iii) a cooling flavor.
- 40. The coated chewing gum of claim 39 wherein the concentration of cooling flavor is reduced from conventional concentrations such that harsh
   10 notes are substantially reduced or eliminated.
  - 41. A chewing gum composition comprising:
    - a) about 5% to about 95% gum base;
    - b) about 5% to about 95% bulking and sweetening agent;
- 15 and
  - c) about 0.1 to about 10% flavoring agent wherein the flavoring agent comprises an acyclic carboxamide and one or more other physiological cooling agents.
- 20 42. The chewing gum composition of claim 41 wherein the acyclic carboxamide comprises N-2,3-trimethyl-2-isopropyl butanamide.
  - 43. The chewing gum composition of claim 42 wherein the one or more other physiological cooling agents comprises menthyl succinate.

WO 99/13734

PCT/US97/16731

- 44. A chewing gum composition comprising:
  - a) about 5% to about 95% gum base;
  - about 5% to about 95% bulking and sweetening agent;

5

and

- c) about 0.1 to about 10% flavoring agent wherein the flavoring agent comprises an acyclic carboxamide and menthol.
- 45. The chewing gum composition of claim 44 wherein the acyclic carboxamide comprises N-2,3-trimethyl-2-isopropyl butanamide.

10

- 46. The chewing gum composition of claim 45 wherein the flavoring agent further comprises menthyl succinate.
- 47. A method of producing a chewing gum with a physiological cooling agent comprising the steps of:
  - a) mixing a physiological cooling agent comprising an acyclic carboxamide together with a release modification material;
  - b) treating the cooling agent to provide a modified release; and

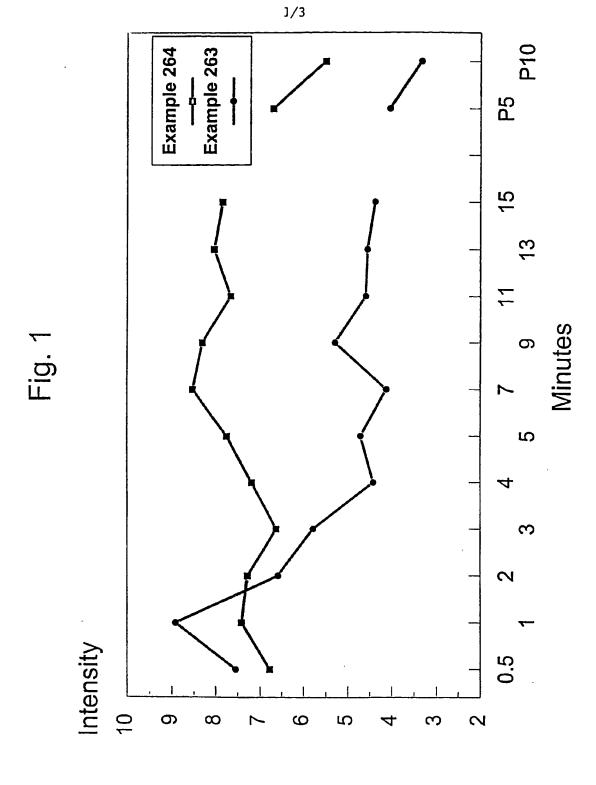
- c) adding a quantity of the modified release cooling agent to a chewing gum composition to provide a cooling agent level in the gum of from about 0.001% to about 1%.
- 48. The method of claim 47 wherein the acyclic carboxamide comprises N-2,3-trimethyl-2-isopropyl butanamide.

PCT/US97/16731

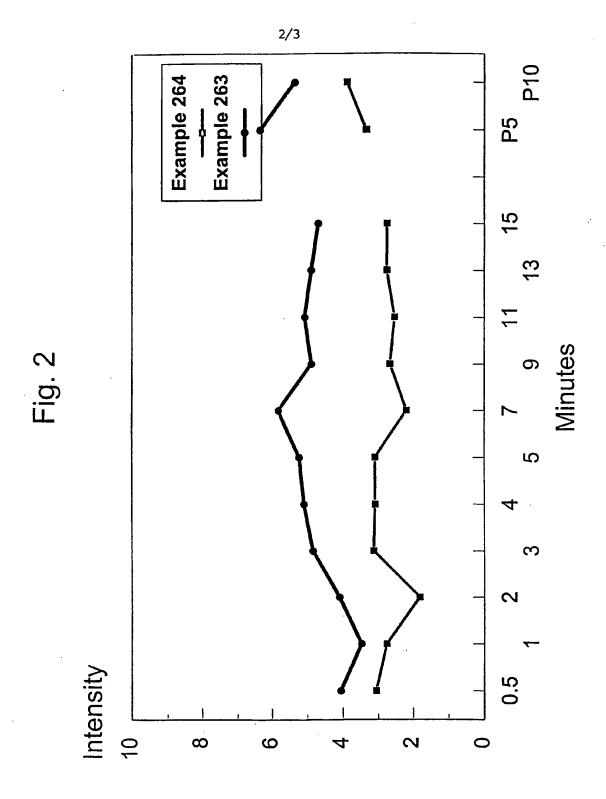
and

15

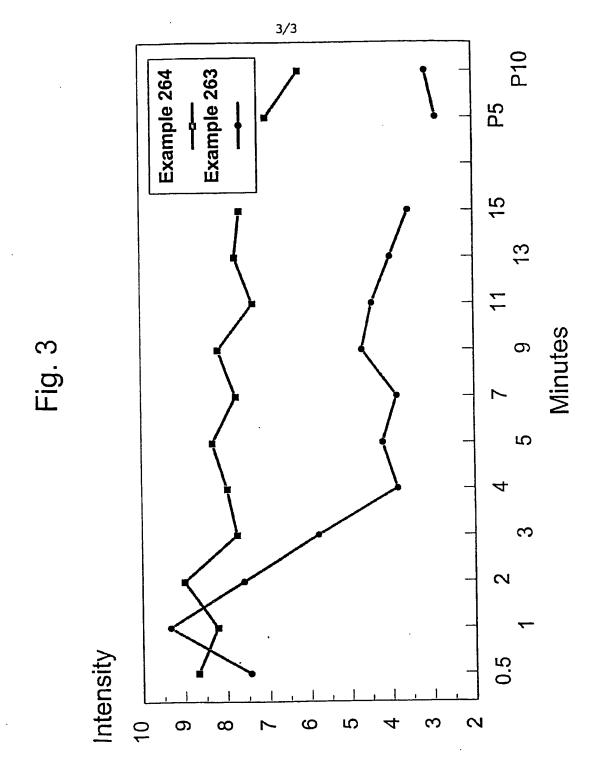
- 49. A chewing gum composition comprising:
  - a) about 5% to about 95% gum base;
  - b) about 5% to about 95% bulking and sweetening agent;
- 5 c) about 0.1% to about 10% flavoring agent wherein the flavoring agent comprises a physiological cooling agent and a spicy hot flavor but is substantially free of menthol and other mint oil components.
- 50. The chewing gum composition of claim 49 wherein the spicy hot flavor is selected from the group consisting of cinnamon, clove, ginger, black pepper, cayenne pepper and combinations thereof.
  - 51. The chewing gum composition of claim 49 wherein the flavoring agent comprises less than 10% of mint components.
  - 52. The chewing gum composition of claim 49 wherein the spicy hot flavor comprises cinnamon and the physiological cooling agent is selected from the group consisting of menthane glycerol ketal, menthyl lactate, menthyl succinate N-substituted p-menthane carboxamide, acyclic carboxamide and mixtures thereof.



**SUBSTITUTE SHEET (RULE 26)** 



WO 99/13734 PCT/US97/16731



SUBSTITUTE SHEET (RULE 26)

## INTERNATIONAL SEARCH REPORT

International application No. PCT/US97/16731

IPC(6) :A23 US CL :424	FICATION OF SUBJECT MATTER 3G 3/30; A23L 1/22, 2/56; A61K 9/68 1/48; 426/3, 5, 6, 534, 536, 537, 538					
According to International Patent Classification (IPC) or to both national classification and IPC						
	SEARCHED					
Minimum docum	mentation searched (classification system followe	d by classification symbols)				
U.S. : 424/	/48; 426/3, 4, 5, 6, 534, 536, 537, 538 					
Documentation s	searched other than minimum documentation to the	e extent that such documents are included	in the fields scarched			
Electronic data t	base consulted during the international search (ne	ame of data base and, where practicable,	, search terms used)			
C. DOCUM	ENTS CONSIDERED TO BE RELEVANT					
Category •	Citation of document, with indication, where ap	propriate, of the relevant passages	Relevant to claim No.			
	S 5,372,824 A (RECORD ET AL) 13 e entire document.	B December 1994 (13.12.94),	49-51			
Y	e entire document.		1-48, 52			
	O 96/17524 A (LUO) 13 June 1 ocument.	1996 (13.06.96), see entire	1-7, 47, 48			
	Y US 5,326,574 A (CHAPDELAINE ET AL) 05 July 1994 1-7, 47, 48 (05.07.94), see entire document.					
I.	S 4,724,151 A (MANSUKHANI 19.02.88), see entire document.	ET AL) 09 February 1988	1-7, 47, 48			
	S 5,128,155 A (SONG ET AL) 07 Jul ocument.	y 1992 (07.07.92), see entire	6			
X Further do	ocuments are listed in the continuation of Box C.					
"A" documen	estegories of cited documents: at defining the general state of the art which is not considered particular relevance	"T" later document published after the inte date and not in conflict with the appli the principle or theory underlying the	cation but cited to understand			
,	ocument published on or after the international filing date	"X" document of particular relevance; the considered novel or cannot be consider				
cited to	at which may throw doubts on priority claim(s) or which is establish the publication date of another citation or other	when the document is taken alone	·			
special reason (as specified)  "Y"  document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art						
	"P" document published prior to the international filing date but later than "&" document member of the same patent family the priority date claimed					
Date of the actual completion of the international search  10 DECEMBER 1997  Date of mailing of the international search report  1 4 JAN 1998						
Commissioner of Box PCT	ng address of the ISA/US  f Patents and Trademarks	Authorized offices Buc ARTHUR L. CORBIN	tion			
Washington, D.C	C, 20231 (703) 305-3230	Telephone No. (703) 308-3850				

## INTERNATIONAL SEARCH REPORT

International application No.
PCT/US97/16731

0.40		PCT/US97/167	31
	ation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the releva	nt passages	Relevant to claim N
Y	US 4,317,838 A (CHERUKURI ET A) 02 March 1982 see entire document.	(02.03.82),	12-40
Y	US 5,376,389 A (REED ET AL) 27 December 1994 (27 see entire document.	7.12.94),	12-40
Y	US 4,029,759 A (HUMBERT ET AL) 14 June 1977 (14 see entire document.	.06.77),	12-40
c	US 4,153,679 A (ROWSELL ET AL) 08 May 1979 (08. entire document.	05.79), see	41-48